Final Technical Report

For

Mercury Removal in a Non-Thermal, Plasma-Based Multi-Pollutant Control Technology for Utility Boilers

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Abstract

Powerspan has conducted pilot scale testing of a multi-pollutant control technology at FirstEnergy's Burger Power Plant under a cooperative agreement with the U.S. Department of Energy. The technology, Electro-Catalytic Oxidation (ECO), simultaneously removes sulfur dioxide (SO_2), nitrogen oxides (NO_x), fine particulate matter ($PM_{2.5}$) and mercury (Hg) from the flue gas of coal-fired power plants. Powerspan's ECO® pilot test program focused on optimization of Hg removal in a 1-MWe slipstream pilot while maintaining greater than 90% removal of NO_x and 98% removal of SO_2. This Final Technical Report discusses pilot operations, installation and maintenance of the Hg SCEMS instrumentation, and performance results including component and overall removal efficiencies of SO_2, NO_x, PM and Hg from the flue gas and removal of captured Hg from the co-product fertilizer stream.

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1.0 Introduction

Powerspan Corp. (New Durham, NH) has developed an integrated air pollution control technology that achieves major reductions in emissions of NO_x (90%), SO_2 (98%), fine particulate matter (95%), and mercury (80 to 90%) from the flue gas emitted by coal-fired power plants. The patented technology, named Electro-Catalytic Oxidation (ECO), also reduces emissions of air toxic compounds such as arsenic and lead as well as acid gases such as hydrochloric acid (HCl). An ECO pilot unit is installed at FirstEnergy's Burger Power Plant and has been in operation since February 2002 under a $2.8 million cooperative agreement with the U.S. DOE (NETL). The pilot treats 1500 to 3000 scfm of flue gas drawn from the Burger Plant's Unit 4 or 5 boilers.

In commercial operation the ECO process is to be installed downstream of a power plant's existing electrostatic precipitator or fabric filter. It treats flue gas in four steps to achieve multi-pollutant removal. In the first process step a barrier discharge reactor oxidizes gaseous pollutants to higher oxides. For example, nitric oxide is oxidized to nitrogen dioxide and nitric acid, a small portion of the sulfur dioxide is converted to sulfuric acid, and mercury is oxidized to mercuric oxide. Following the barrier discharge reactor is an ammonia based scrubber, which removes unconverted sulfur dioxide and the nitrogen dioxide produced in the barrier discharge, creating an ammonium sulfate – ammonium nitrate solution. A wet electrostatic precipitator (WESP) follows the scrubber. It, along with the scrubber, captures acid aerosols produced by the discharge reactor, fine particulate matter and oxidized mercury. The WESP also captures aerosols generated in the scrubber. Finally, liquid effluent produced by the scrubber contains dissolved ammonium sulfate and nitrate salts, along with Hg and captured particulate matter. It is sent to a co-product recovery system, which includes filtration to remove ash and activated carbon adsorption for Hg removal. The treated co-product stream, free of Hg and ash, can be processed to form ammonium sulfate nitrate (ASN) fertilizer in crystal, granular or liquid form.

A simplified flow diagram for the ECO process in a commercial installation is shown in Figure 1. The figure shows the process equipment connected to a plant's existing ductwork. A booster fan moves flue gas through the barrier discharge reactor then the absorber/WESP tower. Aqueous ammonia and water is added to the upper loop of the absorber to maintain scrubbing pH and density. Liquid is transferred from the upper loop to the lower loop to maintain upper loop scrubbing conditions. Particulate matter and aerosols captured in the WESP are also drained to the lower loop through condensation in the WESP and periodic washing. Evaporation of water in the lower loop concentrates the liquid to a near saturated ammonium sulfate – nitrate (ASN) solution. The co-product stream is then drawn from the lower loop, as shown in the figure, and process to produce commercial grade fertilizer. Water removed from the co-product is then returned to the ECO process.

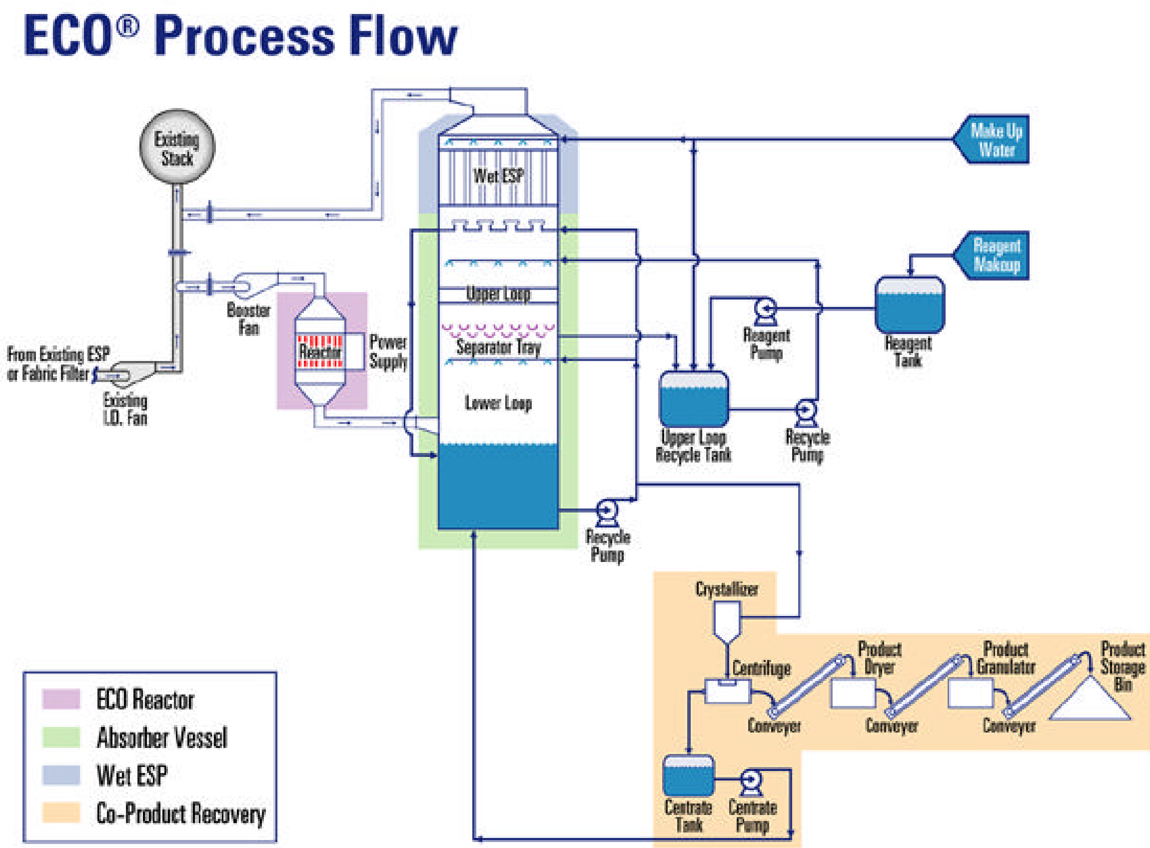


Figure 1 ECO Process Flow Diagram

Laboratory testing had shown the ability of the ECO process to capture mercury in simulated flue gas streams. The work conducted under this cooperative agreement was intended to demonstrate mercury removal in power plant flue gas while maintaining high removal levels of NO_x, SO_2, particulate matter, acid gases and air toxic compounds. The project utilized the ECO pilot constructed at FirstEnergy's Burger Power Plant near Shadyside, Ohio.

2.0 Executive Summary

Powerspan's ECO® pilot test program focused on optimization of Hg removal in a 1-MWe slipstream pilot while maintaining greater than 90% removal of NO_x and 98% removal of SO_2. Although only limited data was acquired to optimize conditions for elemental mercury oxidation, the pilot test results to date show ECO to be a multi-pollutant control process capable of achieving high removal of Hg, NO_x, SO_2, and particulate matter. Successful pilot testing has led to the construction of a 50 MW commercial demonstration unit at FirstEnergy's Burger Power Plant.

Operation of the mercury SCEMs used in the pilot testing required extensive experimentation and troubleshooting throughout the project. After exhaustive testing it was determined that the sampling system provided with the PS Analytical instruments was unable to perform adequately in the heavy ash environment that existed in pilot's inlet flue gas stream, drawn from the inlet of the Burger Plant's electrostatic precipitator. The reactive nature of the flue gas ash with mercury, combined with the inability to adequately clean the sample filter between sample events, skewed measurements of elemental and oxidized mercury. In addition, frequent sample contamination due to inadequate filtering of ash led to unreliable measurement of gas-phase mercury and required replacement of the inlet sample probe with an inertial separation probe from Apogee Scientific. The inertial separation probe improved measurement of elemental and oxidized mercury in the inlet flue gas. As a result of the problems encountered with speciated mercury measurement in the ash laden environment of the pilot's inlet flue gas, a new duct configuration was installed for the pilot. The new ductwork provided flue gas from the outlet of the Burger Plant's Unit 4 electrostatic precipitator, reducing the ash loading on the pilot system by an order of magnitude, to levels expected for commercial ECO installations.

Measurement of mercury in the ECO process fluids was successfully accomplished throughout the project after modification of standard measurement methods. The modifications were required to eliminate effects of non-metallic compounds in the sample matrix of the ECO process streams.

Mercury removal from the ECO process fluids prior to crystallizing the ammonium sulfate nitrate (ASN) fertilizer was also successfully accomplished during this project. The mercury was removed from pilot effluent using Mersorb LW. It was also shown that KeyleX, a sulfonated chelating ion exchange resin also removes mercury from pilot effluent. More testing is required to determine which of the mercury removal adsorbents is most cost effective for commercial application.

Pilot testing of the ECO process and its economics point out several advantages to the ECO system that make it attractive for pollutant control in coal combustion produced flue gas streams. They include:

1. Performance of the ECO technology at the pilot scale continues to show its ability to remove 90% of NO_x, 98% of SO_2, 80% to 90% of Hg, and 99.9% of fine particles that are less than 10 µm in diameter.
2. The ECO system significantly reduces emissions of NO_x, SO_2, $PM_{2.5}$, and Hg in an integrated system, thereby minimizing the need for additional capital investment in other pollution control equipment.
3. The ECO system produces a commercially salable, ammonium sulfate-nitrate fertilizer byproduct that reduces operating costs and avoids landfill disposal of waste.
4. Capital costs for the ECO system are estimated to be approximately $250 per KW including balance of plant modifications. Levelized O&M costs are estimated to be 2.0 to 2.5 mils/KW-hr.
5. The ECO system minimizes the plant retrofit requirements and plant down time for installation, since it provides multi-pollutant control with a single, tie-in installation.
6. ECO equipment has a much smaller footprint than conventional control technologies, facilitating its installation on space-constrained sites that are typical of the existing coal-fired electricity generating fleet.

3.0 ECO Technology Overview

The ECO (Electro-catalytic Oxidation) system is a four-stage pollution control process that integrates several technologies to remove high levels of the primary air pollutants generated by coal-fired power plants. The four stages of ECO technology are:

Stage 1: A dielectric barrier discharge reactor that oxidizes NO and elemental Hg

Stage 2: An absorber that removes SO_2 and NO_2

Stage 3: A wet electrostatic precipitator used to collect aerosols and fine particles

Stage 4: A co-product treatment system for removal of Hg and ash from the liquid co-product stream prior to produce solid commercial grade fertilizer

A more complete description of each one of the ECO stages is given below.

<u>Stage 1: ECO Reactor</u> - *Oxidation of pollutants*

A dielectric barrier discharge (DBD) reactor is used to initiate the ECO process chemistry. A dielectric barrier discharge reactor (DBD) uses non-thermal plasma to generate high energy electrons (~5eV) that have an energy ideal for creating hydroxyl (OH) radicals and atomic oxygen (O). The mechanism for the formation of these radicals [1] is through the collision of electrons with water and oxygen molecules present in the flue gas as shown in reactions (1) through (3).

$$O_2 + e^- \rightarrow O + O + e^- \quad (1)$$

$$H_2O + e^- \rightarrow OH + H + e^- \quad (2)$$

$$O + H_2O \rightarrow 2OH \quad (3)$$

The hydroxyl radicals and atomic oxygen are the foundation of the oxidation reactions initiating the ECO process chemistry. The reactions shown in (4) through (7) are the pathways that oxidize Hg to mercuric oxide (HgO), SO_2 to sulfuric acid (H_2SO_4), and NO_x to nitric acid (HNO_3) and nitrogen dioxide (NO_2).

$$OH + SO_2 \rightarrow HSO_3 \quad (4)$$

$$HSO_3 + O_2 \rightarrow HO_2 + SO_3 \quad (5)$$

$$SO_3 + H_2O \rightarrow H_2SO_4 \quad (6)$$

$$NO + HO_2 \rightarrow NO_2 + OH \quad (7)$$

$$O + NO \rightarrow NO_2 \quad (8)$$

$$OH + NO_2 \rightarrow HNO_3 \quad (9)$$

$$O + Hg \rightarrow HgO \quad (10)$$

The above reactions are only a few of the many reactions that are initiated by the discharge. Others, including the oxidation of SO_2 by O atoms, can also lead to the production of acids from the flue gas pollutants. A detailed description of DBD processing of NO_x in a flue gas stream can be found in References [2] through [4]. Combined DBD processing of NO_x and SO_2 is discussed in References [5] through [8]. The removal of NO from the flue gas stream by ECO is driven by the ability of the DBD to convert NO to NO_2 and HNO_3. Once in these more soluble forms, the ECO scrubber chemistry and wet electrostatic precipitator will capture both.

Although the actual conversion varies with inlet conditions, Figure 2 shows the typical conversion of NO and SO_2 in Powerspan's barrier discharge reactor as a function of the electrical energy supplied to the reactor. The inlet NO_x concentration for this data is 0.4 lb/MMBtu. NO conversion shown in the figure is the portion of flue gas NO converted to either NO_2 or HNO_3. The curve labeled NO_x conversion represents the portion of NO converted to HNO_3, so that the difference between NO and NO_x conversion is the percentage of incoming NO converted to NO_2. SO_2 conversion represents the percentage of flue gas SO_2 converted to SO_3 and H_2SO_4.

In addition to oxidation of NO and SO_2, the DBD reactor oxidizes elemental Hg. Elemental mercury has a low solubility in aqueous solutions, making it difficult to remove from flue gas streams. On the other hand, it has been repeatedly shown in FGD systems that oxidized mercury can be captured by scrubber solutions because of its increased solubility. Therefore, oxidizing elemental mercury in the flue gas stream increases the Hg removal ability of scrubbers. Oxidized mercury will pass through the DBD reactor unaffected.

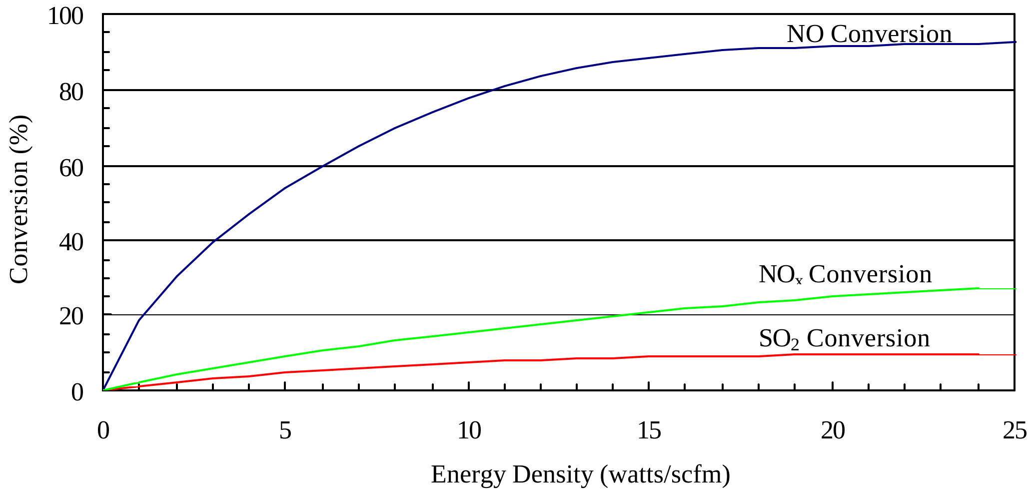


Figure 2: Dielectric Barrier Discharge Conversion for 0.4 lbm/MMBtu Inlet NO_x

<u>Stage 2: Scrubber</u> - *Collection of SO_2 and NO_2*

An ammonia scrubber is used in the ECO process in order to achieve high removal efficiencies of NO_x and SO_2 while reducing the power requirements of the barrier discharge reactor. Flue gas entering the scrubber is first quenched to saturation. The gas then enters a scrubbing stage, which captures both SO_2 and NO_2. Ammonia is utilized in order to scrub SO_2 at a high rate and to produce compounds capable of reducing NO_2 to nitrogen. Ammonia also neutralizes the acids (HNO_3 and H_2SO_4) created in the barrier discharge reactor and produces ammonium sulfate nitrate (ASN), a useful fertilizer byproduct, from the acids and scrubbed SO_2. The synergy between SO_2 scrubbing and capture of NO_2 produced from NO_x in the barrier discharge reactor results in a system with the ability to achieve high levels of NO_x and SO_2 removal.

The scrubbing chemistry starts with ammonia in aqueous solutions producing ammonium and hydroxyl (base) ions as shown in reaction (11). Reactions (12) through (14) show the absorption of SO_2 into aqueous solution producing sulfurous acid (H_2SO_3), bisulfite (HSO_3^-), sulfite (SO_3^{2-}) and hydronium (H_3O^+) ions.

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \qquad \textbf{(11)}$$

$$SO_2 + H_2O \leftrightarrow H_2SO_3 \qquad \textbf{(12)}$$

$$H_2SO_3 + H_2O \leftrightarrow HSO_3^- + H_3O^+ \qquad \textbf{(13)}$$

$$HSO_3^- + H_2O \leftrightarrow SO_3^{2-} + H_3O^+ \qquad \textbf{(14)}$$

The hydroxyl and hydronium ions react to maintain a neutral pH, driving the SO_2 absorption reactions (12) through (14) to produce sulfite.

$$OH^- + H_3O^+ \leftrightarrow H_2O \qquad \textbf{(15)}$$

Combining reactions (11) through (14) yields the overall SO_2 scrubbing reaction (16)

$$2NH_3 + SO_2 + H_2O \rightarrow 2NH_4^+ + SO_3^{2-} \qquad \textbf{(16)}$$

Sulfite produced by reaction (16) reacts with NO_2 through oxidation-reduction reaction (17) reducing NO_2 to nitrogen while oxidizing sulfite to sulfate:

$$2SO_3^{2-} + NO_2 \rightarrow SO_4^{2-} + \tfrac{1}{2}N_2 \qquad \textbf{(17)}$$

From reactions (13) through (17), the overall reactions for SO_2 and NO_2 (18) and (19) are shown in Figure 3.

$$2NH_3 + SO_2 + H_2O \rightarrow (NH_4)_2SO_3 \qquad \textbf{(18)}$$

$$2(NH_4)SO_3 + NO_2 \rightarrow 2(NH_4)_2SO_4 + \tfrac{1}{2}N_2 \qquad \textbf{(19)}$$

The ratio of SO_3^{2-} to HSO_3^- in solution (14) is determined by solution pH. Both compounds are reported to scrub NO_2, but the rate of reaction between NO_2 and SO_3^{2-} is reported to be approximately forty times faster than that of NO_2 with HSO_3^-. [1] Therefore, pH control of the scrubbing solution through addition of ammonia is essential to ensure that an adequate SO_3^{2-} concentration is maintained for high NO_2 scrubbing rates. From reactions (18) and (19) it can be seen that a minimum of two moles of SO_2 are required for each mole of NO_2 reduced to N_2. However, the sulfite needed for NO_2 reduction can also be consumed by O_2 in the flue gas, effectively increasing the ratio of SO_2 to NO_2 required for NO_2 scrubbing:

$$2SO_3^{2-} + O_2 \rightarrow 2SO_4^{2-} \qquad \textbf{(20)}$$

Oxidation of sulfite to sulfate results in a minimum ratio of SO_2 to NO_x of 3 in order to maintain a sulfite concentration adequate to scrub NO_2 to acceptable levels.

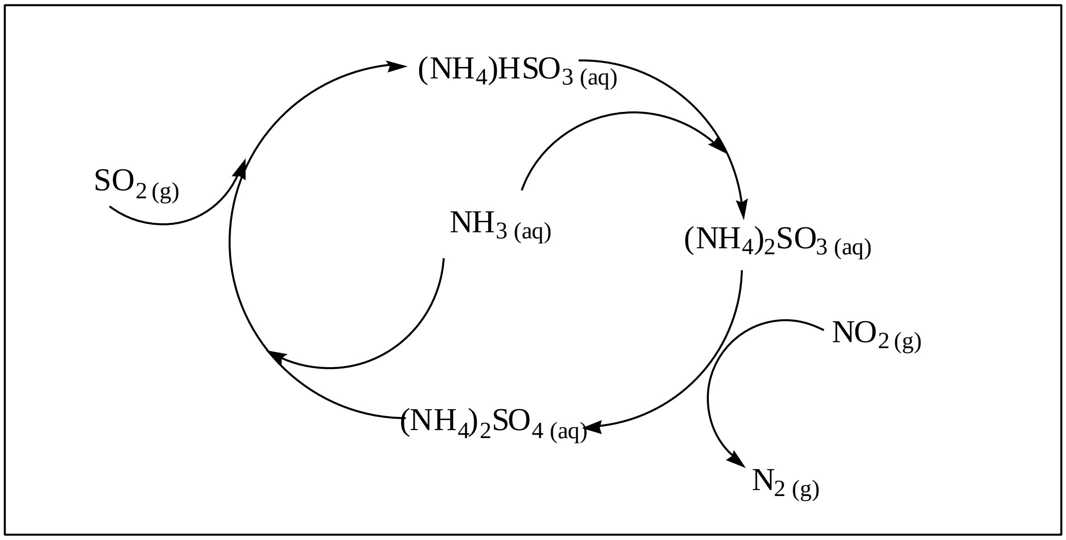


Figure 3 Pathway for NO_2 scrubbing with Sulfite

Compounds produced in the scrubbing of SO_2 and NO_2 can also result in the reduction of oxidized mercury (Hg^{2+}) to elemental mercury (Hg^0), reversing the oxidation process accomplished by the barrier discharge reactor. Understanding the extent to which the reduction reactions occur and, if necessary, developing a means to retard the reaction was a focus of laboratory investigation and the pilot test program. Solutions to the problem of mercury reduction included altering the conditions of the scrubber chemistry to retard the rate of reduction and treatment of the scrubber solution to remove mercury. The treatment process must keep the steady state mercury concentration low enough to substantially diminish the rate of mercury reduction.

Stage 3: Wet ESP - *Collection of acid aerosols and fine particles*

Flue gas exiting the ammonia scrubber contains oxidized mercury and fine particulate matter. It will also contain aerosols generated in the barrier discharge and ammonia scrubbing process steps (NH_4HSO_4, NH_4NO_3, NH_4Cl). These materials are captured in a wet electrostatic precipitator (WESP) and returned to the scrubbing solution. A WESP is efficient at collection of aerosols and fine particulate matter since there is no mechanism to cause particle re-entrainment. In a WESP, liquid flows down the collecting plate to remove captured materials from the plate. The advantages of WESPs include: a) water flow prevents particle re-entrainment, which would limit collection efficiency; b) the water layer does not limit corona current; and c) the improved collection characteristics permit high gas velocities, limiting the equipment size required for collection.

Stage 4: Co-Product Treatment System - *Production of commercial-grade fertilizer*

Ammonium sulfate and nitrate created in the ECO process can be treated and used as a commercial fertilizer. Solids in the scrubber bleed stream, consisting of ash and insoluble metal compounds, are removed by filtration. The stream is then pumped through an activated carbon adsorption bed. The activated carbon used in ECO is produced by Nucon International (Columbus, OH) and sold under the name of Mersorb®. Mercury reacts with the sulfur impregnated activated carbon and is retained in the adsorbent bed while the liquid continues through the bed. Spent mercury-laden activated carbon is replaced and disposed of as a hazardous waste. It is estimated that the variable cost of mercury removal with activated carbon is $733 per pound of mercury, including the media and disposal.

Liquid substantially free of mercury and ash can be used as a fertilizer directly or sent to a crystallizer in which moisture is driven off to produce crystals of well-defined size, strength, and composition. The crystals may be usable as fertilizer in the form produced by the crystallizer, or may be processed to further reduce the moisture of the crystals or to agglomerate the crystals into granules.

The co-product treatment system uses standard fertilizer crystallization and granulation processes and equipment. All processing can be done on-site, or the crystalline material can be generated on site and then shipped to a fertilizer processing plant.

4.0 ECO Pilot Overview

The ECO pilot system, constructed at FirstEnergy's Burger Power Plant, was in operation for four years prior to this cooperative agreement with DOE to support development of the ECO technology and is shown in Figure 4. It was modified at the beginning of 2002 to incorporate the ammonia based NO_2 and SO_2 scrubber and its associated liquid handling equipment. A decision was made prior to the modification to keep the unit in a horizontal configuration, as opposed to the vertical configuration to be commercially deployed. The decision was based on a desire to minimize the time required to complete modifications and investigations of the process chemistry in actual flue gas.

The ECO pilot was supplied with flue gas from the Burger Power Plant's units 4 or 5, drawn from the inlet to the plant's electrostatic precipitators. Each unit is rated for 156 MWe and the units fired a varying mixture of fuels throughout the project. Typically, the fuel consisted of a blend of eastern bituminous and sub-bituminous coals, with periodic firing of petroleum coke.

Upon exiting the Powerspan dry ESP, flue gas entered a multi-tube, coaxial cylinder barrier discharge reactor shown in Figure 5. High voltage applied to the center electrodes of the discharge reactor created the non-thermal plasma that formed radicals leading to oxidation of gaseous pollutants. The ECO pilot reactor was capable of delivering up to 100 KW of electrical discharge energy to the gas.

The ammonia scrubber, shown in Figure 6(a), followed the barrier discharge reactor and was in an absorber vessel consisting of three packed sections in a cross flow configuration. The first section cooled and saturated the flue gas. It was four feet deep in the direction of gas flow. Next was a six-foot scrubbing section to remove SO_2 and NO_2. Following the scrubbing section was a six-inch packed section that absorbed gaseous ammonia exiting the scrubbing section.

Gas exiting the absorber vessel entered a horizontal, three-field WESP shown in Figure 6(b). Each field was thirty inches deep in the direction of gas flow. The collecting plates were washed periodically, and the liquid effluent was sent back to the ammonia scrubber section.

Due to excessive ash loading through the ECO system and the subsequent problems that were introduced, the configuration of the pilot was changed such that the slipstream of flue gas was drawn from downstream of the Burger plant's Unit 4 dry electrostatic precipitators. After this configuration change, the cyclone separator was removed from the gas path and the pilots' dry electrostatic precipitator fields were shut off to make the pilot flue gas representative of what would be seen in a commercially deployed ECO installation. This pilot slipstream configuration change was implemented in July of 2003 and run until December 2003

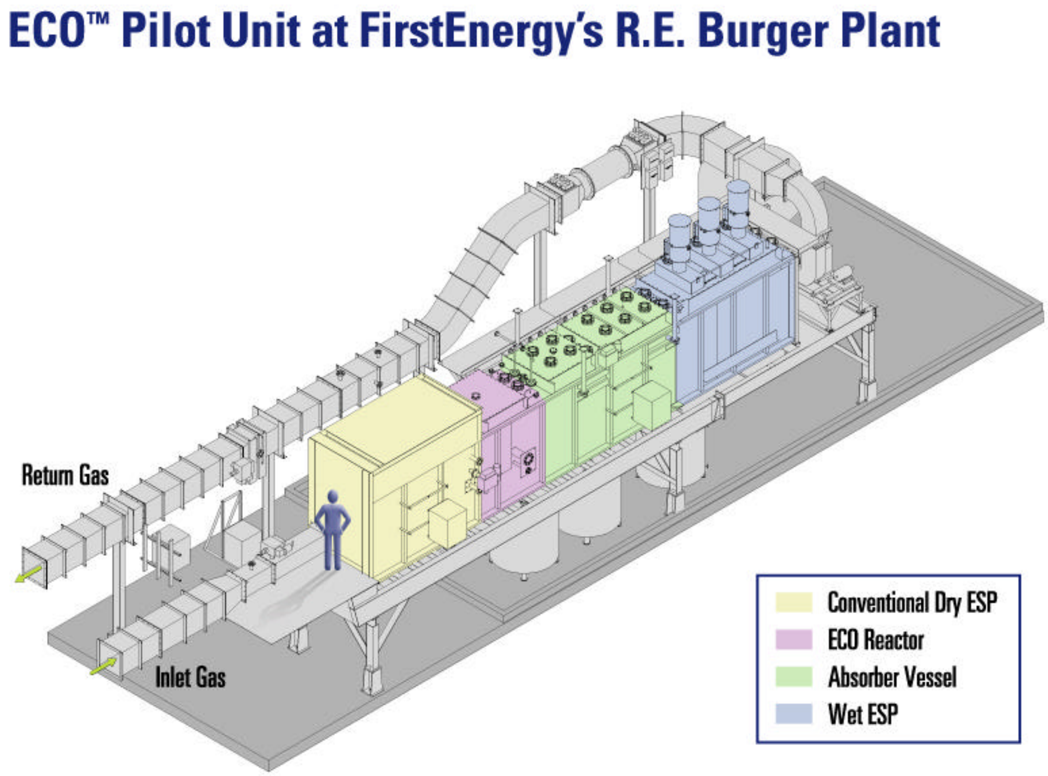


Figure 4: Layout of the ECO Pilot at FirstEnergy's Burger Plant

(a) (b)

Figure 5: (a) ECO Pilot DBD Reactor at the R.E. Burger Generating Station, (b) Discharge looking down the center of one tube

(a) (b)

Figure 6 (a) Top of ECO pilot unit ammonia scrubber, (b) ECO pilot horizontal WESP

A seven-man crew operated the ECO pilot on a 24 hour per day basis. Continuous emissions monitoring was accomplished at the flue gas inlet to and exit from the pilot. The CEMS system measured the concentration of SO_2, NO_x, O_2, H_2O, CO_2, CO and NH_3. Outlet flue gas flow and opacity were also measured continuously. Temperatures, flow rates, pH of all liquid flow streams, and pressure drop across all process units were also measured. Mercury concentration in the pilot flue gas was measured using PS Analytical's Sir Galahad semi-continuous mercury CEM systems. Two systems were installed to provide simultaneous, near real time measurement at the pilot inlet and outlet as well as at other selected locations. The systems reported elemental and total gas phase mercury concentrations and are discussed later in this report. In all, over 175 parameters were continuously recorded by automatic data logging equipment.

4.1 Flue Gas Supply and Ash Removal

The coal burned at FirstEnergy's Burger Power Plant is typically a blend of eastern bituminous and subbituminous coal. The blend varies during normal operation and produces a high percentage of oxidized mercury compared to elemental mercury in the flue gas and a reactive ash. The implications for testing the ECO process on this coal blend are described below.

The pilot, in its initial configuration, drew a slipstream of gas from the Burger Plant's Unit 4 or 5, upstream of the unit's ESP. The gas was returned to the unit at the Burger plant ESP inlet. The gas flow into the pilot, ranged from 1500 to 3000 scfm (standard cubic feet per minute), and passed through a small cyclone separator and two dry ESP fields, each four feet in length. The cyclone separator and ESP in series were used to try to reduce the ash content of the flue gas to a level similar to that expected after a plant's ESP or fabric filter, which is the location for full-scale ECO installations.

Since the pilot flue gas was drawn prior to the plant's ESP and the pilot had minimal ash removal ability, ash loading measurements were made at the inlet of the ECO system and compared to the Burger Plant's reported ash loading on the outlet of the ESP. The results are shown in Figure 8; the red line in the figure is the measured ash loading on the outlet of the Burger Plant's ESP; the green triangles represent ash loading as measured by Air Compliance Testing at the inlet of the ECO process using EPA Method 5: (Determination of Particulate Matter Emissions from Stationary Sources). The blue line shows the results of measurements made by Powerspan using an ash sampler at the inlet of the ECO process. The Powerspan ash sampler reports low compared to the Air Compliance Testing results obtained at the same time. However, the Powerspan ash sampler was designed only to give an indication of the day-to-day variations in ash loading at the inlet of the ECO pilot rather than as an isokinetic sampling system. The measurements show that the ash loading is consistently and substantially higher at the inlet of the ECO system than is present at the outlet of the Burger Plant dry electrostatic precipitator. The high ash loading presented problems for establishing an elemental mercury concentration above the level native to the flue gas.

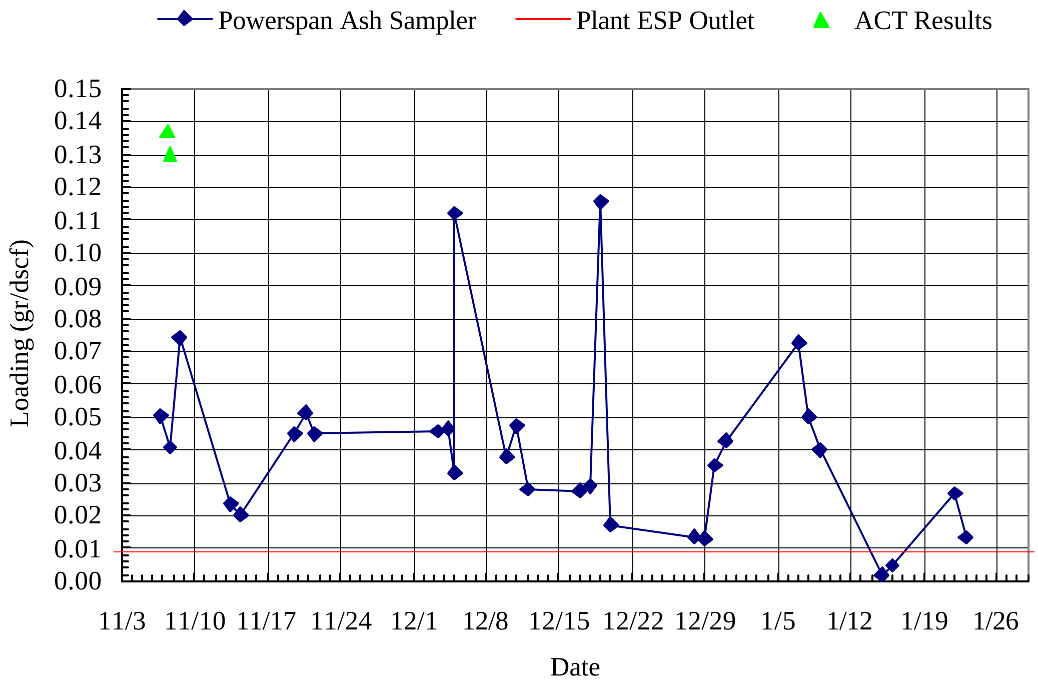


Figure 8: Ash loading measurements made at the ECO system inlet.

After many attempts to perform testing in the high ash loading environment and since the high ash loading was problematic for measuring mercury and was not representative for commercial installations, a new duct was installed at the pilot in July 2003. The new duct drew flue gas from the outlet of the Burger Plant's ESPs providing an ash loading of 0.01 gr/dscf which is representative of a commercial application.

The reactive ash also provided low elemental mercury concentrations in the flue gas. The typical mercury concentration in the flue gas at FirstEnergy's Burger Power Plant contains a low percentage of its total mercury emissions as elemental Hg. As described in a separate task report, Air Compliance Testing performed Ontario Hydro testing during May 8-10, 2002. Table 1 lists the results from Ontario Hydro Testing of the flue gas at the inlet of the ECO process. The results show the elemental Hg fraction is <3% of the total mercury in the flue gas stream.

Hg Fraction	Concentration (µg/Nm^3)
Particle Bound Hg	0.62
Oxidized Hg	5.81
Elemental Hg	0.16
Total Hg	6.59

Table 1: Results from Ontario Hydro testing of the flue gas at the ECO pilot inlet.

However, to effectively demonstrate the ability of the ECO Process to remove elemental mercury, its concentration was artificially raised through the addition of elemental mercury to the flue gas stream. The mercury addition system used is shown in Figure 9 below.

Elemental mercury was added to the flue gas stream with the addition system by saturating a stream of air with mercury. The saturation is controlled by air flow and the temperature of the mercury condensation vessels. Elemental mercury is vaporized in a mercury evaporation vessel and swept out by the air passing through the vessel. The mercury laden air then enters a series of four vessels in a controlled temperature bath. The gas residence time in the condensing vessels ensures that the gas stream is saturated with mercury at the temperature of the condensing bath. The mercury evaporation vessel temperature was set to ensure that the mercury content of the gas leaving the evaporation vessel is greater than the saturation content for the condensing vessel temperature. Since the air/Hg stream exiting the condensing vessels was above saturation for ambient temperature, the stream was diluted prior to leaving the temperature controlled environment of the addition system to eliminate the need to heat trace the addition line to the injection point.

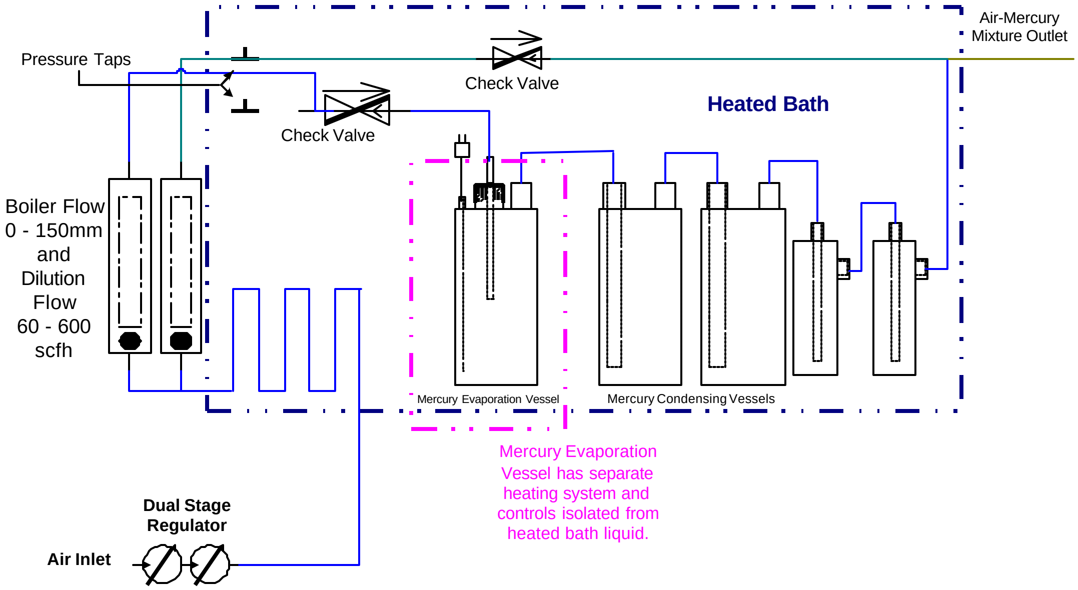


Figure 9: Schematic of the system used to inject elemental mercury into the flue gas.

5.0 Experimental

To understand mercury removal in the ECO process, it was necessary to measure mercury in the flue gas, liquid streams, and solid ASN product. During this project, the flue gas measurements were made using Hg SCEMS equipment, a batch sampler, and Ontario-Hydro measurements done by an outside testing service. A method for measuring Hg in the ECO liquid streams was developed and validated at Powerspan, and the ASN product quality including Hg concentration was measured by an outside laboratory. Each of these areas is addressed in the following sections.

5.1 Mercury Measurements in Flue Gas

One type of Hg SCEMS that has been developed for gas-phase Hg measurement is from PS Analytical (Kent, England). It uses wet chemistry to differentiate elemental mercury from oxidized mercury, and atomic fluorescence for mercury measurement. The system automates sampling, speciation, and mercury measurement to quantify the mercury in flue gas in near real time. Its use at a power plant on real flue gas requires protocols for validation and maintenance to insure reliable data collection.

Although the expectation was that the PS Analytical system was a proven technology ready for operation in power plant conditions, the effort required to obtain measurements with the Hg SCEMS instrumentation turned out to be extensive. It required substantial troubleshooting and modifications to get the instrumentation to a point where it could be used to acquire meaningful data. A detailed discussion of the substantial efforts to develop operating procedures, protocols, troubleshooting, and modifications used to validate the Hg SCEMS instrumentation to be able to obtain reliable, speciated Hg measurements for this program can be found in a separate task report. However, a summary of the mercury instrumentation, measurement techniques, and mercury injection is provided below.

5.1.1 Hg SCEMS Measurements

Instrumentation and Measurement

Since mercury measurement is an essential element in the development of any mercury removal process, a substantial amount of time was spent investigating, selecting, installing, and validating the Hg SCEMS equipment. A brief description of the Hg SCEMs used in this project is presented here. A more comprehensive and detailed description of the Hg SCEMS, the operating protocols, and the problems associated with operating them on a continuous basis can be found in the *Task 1 Technical Report*.

Each Hg SCEM system is composed of two sample probes, two mercury speciation modules, an instrument rack containing a Sir Galahad II mercury analyzer, a stream selector and a CAVkit unit (calibration verification kit). The CAVkit is capable of delivering either zero-air or elemental mercury spiked air to the sample probes for use in troubleshooting the Hg CEMs systems for leaks, contamination and malfunctions. A computer located in the instrument rack controls the Hg SCEM. A simple schematic of a typical system is provided in Figure 10 below.

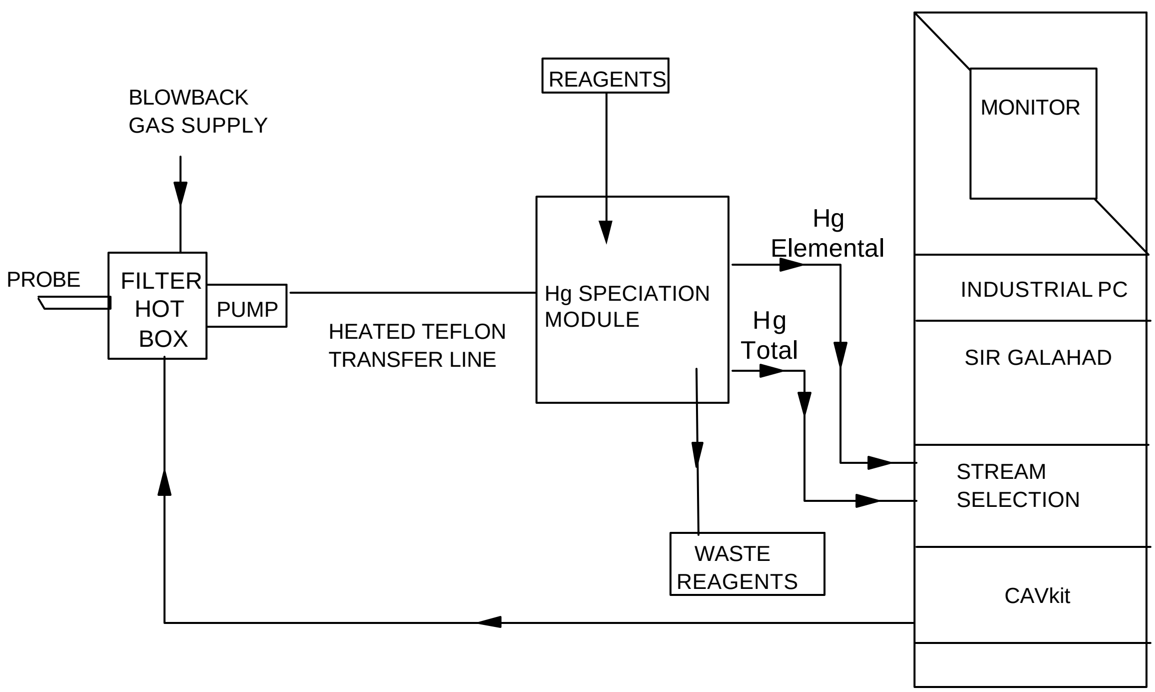
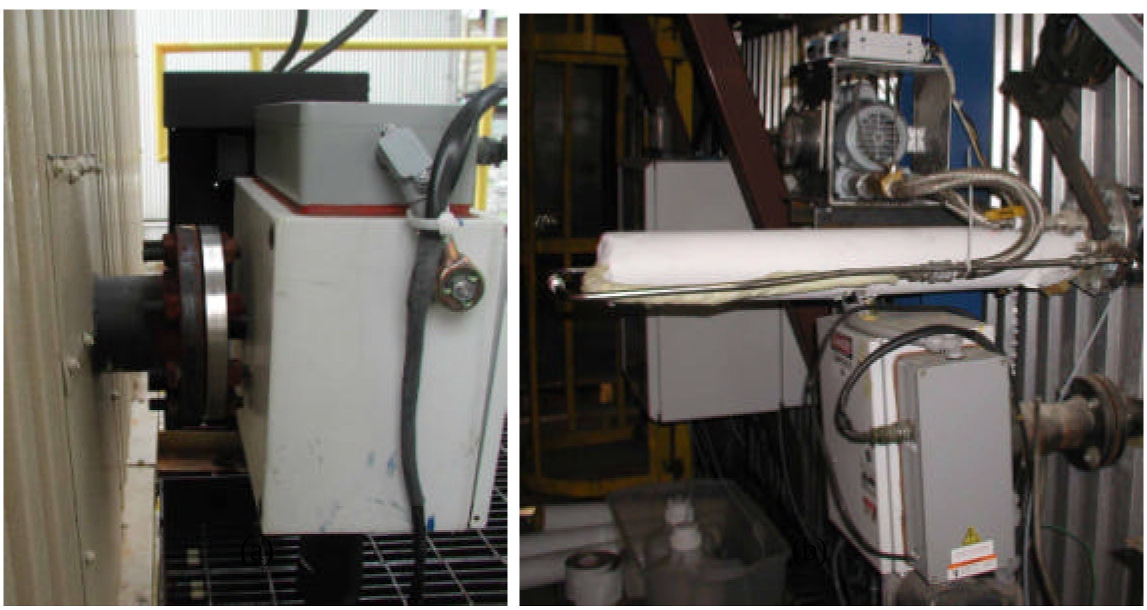


Figure 10: Schematic of a single probe Hg SCEM

A flue gas sample is drawn from the duct through a Teflon stinger and filter using a heated sample pump. The sample is then delivered to an Hg speciation unit with a heated Teflon sample line operated at 400^{o}F to keep the sample gas temperature above the dewpoint and to keep oxidized Hg from adsorbing to the probe surfaces and sample lines. The flue gas sample is maintained at 400 ^{o}F until being treated by the mercury speciation unit, where the sample stream is split into two parts, one for elemental Hg analysis and the other for total Hg analysis. After speciation, heated Teflon sample lines deliver conditioned flue gas to the stream selector to be analyzed in turn by the Sir Galahad analyzer. Sampling and analysis is controlled with software provided by PS Analytical on the computer located in the instrument rack.

During this project, flue gas samples were extracted from the duct using two different sample probe types. The PS Analytical sampling systems were delivered with Baldwin Environmental model 35Hg heated stack filter probes. The probes were installed at four locations in the ECO pilot. However, the inlet probe had numerous problems associated with ash in the flue gas and was replaced for a portion of the project with an Apogee Scientific Quick Silver Inertial Separation (QSIS) probe. The Apogee



(a) (b)

Figure 11: (a) A Baldwin Hg-35 heated sample probe mounted to the inlet duct; (b) QSIS sample probe assembly mounted to the ECO pilot inlet duct

probe was used exclusively at the inlet sampling location. The Baldwin sample probes were used occasionally on the inlet and always on the outlet of the system. A photograph of the Baldwin Hg 35 Probe and the QSIS probe are shown in Figure 11. The spectrometer in the Sir Galahad Analyzer can only measure elemental mercury; therefore it is necessary to use a sample conditioning unit to convert oxidized mercury into elemental mercury. The Hg speciation module and sample conditioner uses wet chemistry to differentiate between elemental and oxidized mercury and removes water from the sample stream to protect the analyzer and maintain the integrity of the sample. A photograph of one of the conditioning units is shown below in Figure 12.

Flue gas is pumped from the sample probes to the sample conditioner using the probe's heated head pump. Flue gas entering the sample conditioner is split into two streams that bubble through two impingers: one for elemental Hg measurement and one for total Hg measurement. Teflon valves are used to control the amount of flow going to each of the channels and the vent. The impingers contain reagents specific to each channel and are filled and emptied using Teflon capillaries and a peristaltic pump. (A detailed description of the reagent chemistry can be found in the Task 1 Technical Report). The flue gas continues through a Peltier Cooler that removes moisture and decreases the dewpoint of the flue gas to 5°C. Before being sent through a heated Teflon sample line to the stream selector and Galahad analyzer, the flue gas passes water slip detectors which are designed to shut off the pumps in the event that liquid is present in the sample stream.

Each instrument rack in the Hg SCEMS contains a 16-channel stream selector, a Sir Galahad II mercury analyzer and a CAVkit unit. A photograph of an instrument rack is shown in Figure 13. The stream selector allows numerous sample streams to be monitored with the same analyzer by switching to a different stream. The stream selector consists of 8 three-way Teflon switching valves and a digital mass flow controller that regulates the flow of flue gas over the Amasil trap in the Sir Galahad II analyzer. Valves are activated using the TTL line from the Sir Galahad. When a stream has been selected for analysis, the corresponding valve is energized using 12-volt dc signal. In addition to sampling from any of four gas streams, the stream selectors are also capable of directing zero air and CAVkit gas to the appropriate sample probe.

Figure 12: Photograph of an installed PSA conditioning unit

Figure 13: Photograph of a PS Analytical Hg SCEMS instrument rack

The Sir Galahad II analyzer uses an atomic fluorescence spectrometer to detect elemental mercury. To make a measurement of elemental mercury, a gold impregnated silica (Amasil) trap is used to adsorb mercury from the flue gas. Using an Amasil trap, the Hg is pre-concentrated to produce a signal that is easily measured and calibrated to by the analyzer. Flue gas is drawn over the trap at a flow rate of 0.5 L/min, which is regulated by the mass flow controller in the stream selector, for an amount of time pre-determined for the expected Hg concentration to be measured. Once sampling is completed, the trap is flooded with argon and the Amasil trap is heated to re-vaporize the mercury and carry it into the analyzer. The elemental Hg is carried past a mercury vapor lamp producing fluorescence. The fluorescence produced is measured by a conventional photomultiplier tube (PMT) creating a signal that is proportional to the concentration of Hg in the sample.

Calibration of the analyzer involves injection of a known amount of mercury vapor onto the gold trap. The calibration is based on the vapor pressure of mercury, which is well known. A measured volume of mercury is withdrawn from the calibration vessel and is injected onto the Amasil trap using a needle and syringe. The Hg is then re-vaporized and carried to the detector as with a flue gas sample, where the peak height or peak area of the response is measured. A calibration curve is generated by plotting the instrument signal against the injected mass of mercury for several mercury concentrations. The PSA software calculates the expected Hg concentration for the calibration based on the temperature and volume used for the calibration spike.

The CAVkit unit is a device for generating elemental mercury vapor that can be sent out to the Hg SCEMS sample probes. The CAVkit gas is used extensively to troubleshooting the Hg CEMs systems for leaks, contamination and malfunctions. To generate elemental mercury, zero air is sent through a small reservoir which contains ~15 grams of elemental mercury adsorbed onto an inert substrate. Changing the temperature of the mercury reservoir varies the elemental Hg output from the CAVkit due to the change in mercury vapor pressure. The CAVkit unit, under normal operating conditions, can generate a maximum of 20 $\mu g/Nm^3$ of elemental mercury at a flow of 16 lpm. The CAVkit is also capable of sending just the zero air to the sample probes that can be used to perform instrument blanks.

Hg SCEMS Troubleshooting

Frequent monitoring and maintenance of the Hg SCEMS components was required in order to keep the instrumentation operating for more than a few hours at any one time. Two complete measurement systems were installed at the ECO pilot, with the ability to sample from four locations in the process. However, due to the extensive efforts to keep the measurement systems operating, sampling was restricted to two locations at any time.

Several significant problems associated with measuring mercury in the flue gas stream were revealed during testing on actual flue gas. These problems can be broken into three types: (i) Hg speciation module, (ii) sample gas extraction, and (iii) hardware failures. Each of these three areas of concern and the steps taken to address these issues are discussed in extensive detail in the *Task 1 Technical Report*.

The Hg Speciation Module is an integral part of the measurement system. Its operation is essential to determine the fraction of elemental and oxidized mercury in the gas phase of the flue gas stream. During operation, many problems were addressed to be able to operate the speciation module to obtain quality data without damaging equipment. These problems included (i) sample flow control, (ii) acid gas removal, (iii) moisture removal, (iv) impinger precipitates, (v) reagent contamination, (vi) reagent refresh rates, and (vii) hardware failures. Most of these difficulties were sufficiently resolved, but only with the expenditure of a considerable amount of time and money.

Testing in an environment with high ash loading has led to (i) difficulties extracting an ash-free sample from the inlet sampling location and (ii) oxidation of elemental mercury with reactive ash. The PS Analytical equipment performed well in the clean flue gas environment at the outlet of the ECO process. However, the ash loading at the inlet of the ECO system, and the ability of the ash to oxidize elemental mercury, proved problematic for accurately determining the concentrations of elemental and oxidized mercury in the flue gas. Extensive efforts to improve the inlet flue gas measurements included testing of multiple sample filters, changing sample probe operating conditions, consulting with industry experts, installation and testing of inertial separation based sampling systems, and installation of ductwork in order to provide flue gas with a reduced ash loading to the ECO pilot. None of these efforts was successful at providing a system and conditions where routine and accurate measurement of the mercury species contained in the flue gas entering the pilot could be made.

5.1.2 Batch Sampling

In addition to the Hg SCEMS instrumentation, a batch sampling method was developed to verify proper operation of the PS Analytical Hg SCEMS instrumentation. An external remote gold trap is used to collect mercury independent of the PSA conditioners and Baldwin probes. By using the remote trap, the measured mercury concentrations can be verified independently of the PSA sample train and on a routine basis.

A schematic of the batch measurement system is shown below in Figure 14. A vacuum pump is used to sample off the QSIS probe in parallel with the PS Analytical instruments. The flue gas is pulled through a series of impingers to speciate the mercury, remove acid gases, and remove moisture. The reagent impinger, the first in the series, is filled with 10% KCl for elemental mercury analysis or 1% $SnCl_2$ in 0.5N H_2SO_4 solution for total mercury analysis. The acid scrubber impinger contains 10 wt% NaOH to scrub

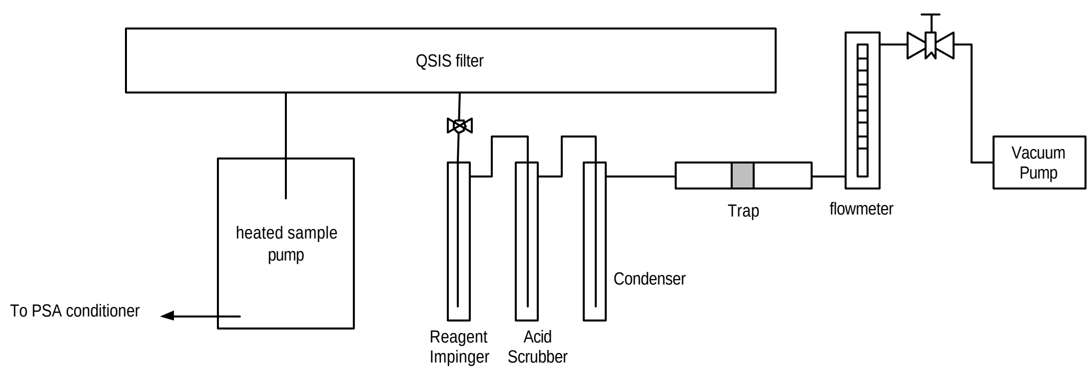


Figure 14: Flow schematic of the batch sampling system attached to the QSIS probe.

out acid gases and finally, the condensing impinger is an empty impinger cooled in an ice bath to remove moisture from the flue gas stream. The flue gas is then passed over a portable Amasil trap to adsorb mercury, similar to what occurs inside the PS Analytical instruments. It is necessary to record gas flow and sample time to convert the mercury mass to a flue gas concentration. Measurement of the captured mercury is made using the external "remote" sample port provided with the PS Analytical Sir Galahad instruments. Once attached to the remote sample port, the analysis of the mercury is the same as described above in Section 4.1.

5.1.3 Elemental Mercury Injection

As discussed earlier, the elemental Hg concentration of flue gas supplied from the Burger plant was too low to demonstrate elemental Hg oxidation. As a result, elemental Hg was added to the flue gas. Figure 15 shows the results of one such addition to flue gas was taken from the inlet of the Burger Power Plant ESP. The expected concentration of Hg(0) was 60 µg/Nm^3. The actual concentrations that were seen were only ~26 µg/Nm^3 of total Hg and ~6.0 µg/Nm^3 of elemental Hg. This represents a recovery of 10% as elemental mercury out of the total mercury added to the flue gas stream. Only 37% of the total mercury added was recovered. The oxidation of the added elemental mercury is attributed to the fly ash in the flue gas stream. Since the flue gas was taken prior to the plants ESP, and the ash removal devices used for the ECO pilot were not as effective as the plant's ESP, the ash loading was approximately 10 times what is expected on the outlet of the ESP. The combination of the amount of ash and its reactive nature significantly complicated the effort to increase the concentration of elemental mercury for testing oxidation of the elemental mercury in the DBD reactor.

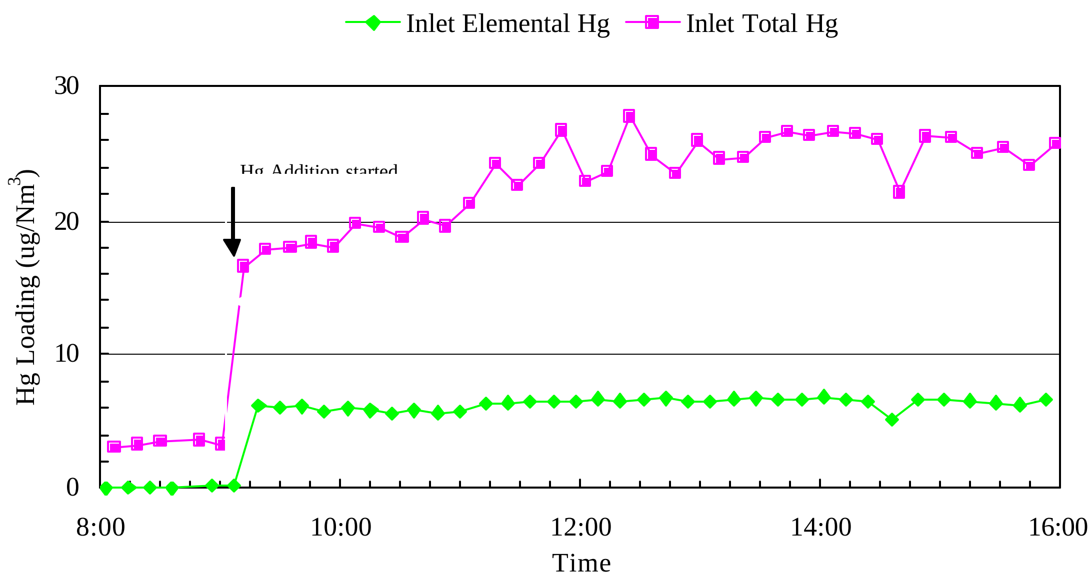


Figure 15 : Addition of elemental mercury vapor to the flue gas

5.1.4 Ash Effects on Elemental Mercury

Clearly, the reactive ash at the R.E. Burger Power Plant could effect the measurement and speciation of mercury. It was also shown in Figure 15 that the ash reacted with injected elemental Hg, decreasing the yield of gas phase mercury, both elemental and oxidized that could be obtained with the addition system. Further investigations were done looking at the ash loading of the system and how it compared to conditions that are expected in a typical installation, such as at the outlet of the Burger Plant's ESP.

With the new duct in place, the ability to add elemental Hg was again tested. The results are shown in Figure 16. For this testing, two elemental Hg levels were chosen.

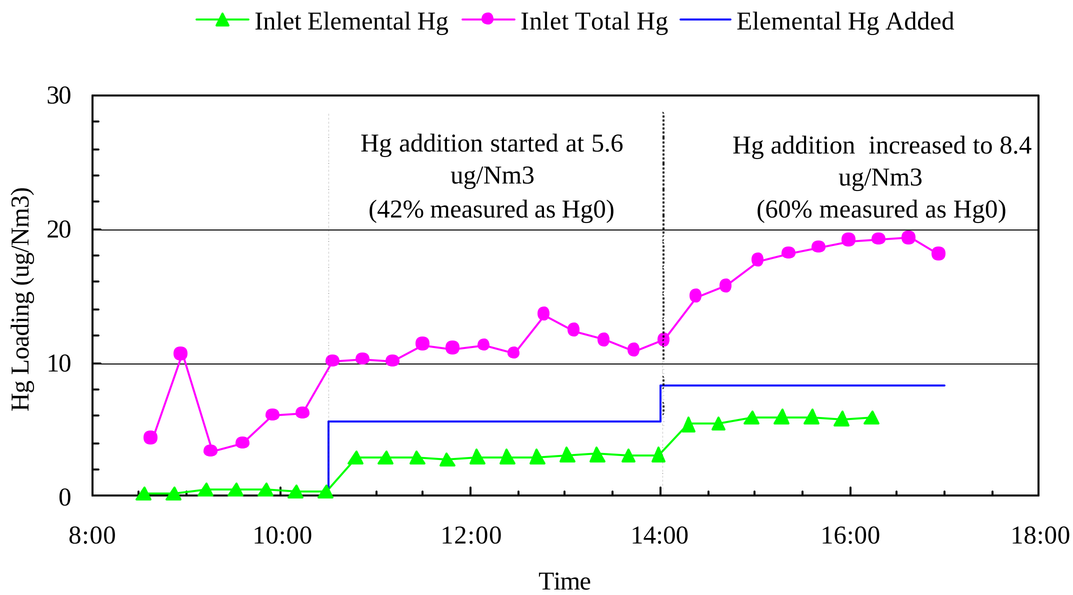


Figure 16: Recovery of Hg^0 after the new duct installation on the outlet of the ESP.

The first was an addition of 5.6 µg/Nm^3 of elemental Hg. The blue line in the figure represents the expected Hg(0) concentration, and the green line represents the amount of elemental mercury actually measured. At this level, 42% of the added mercury reported as elemental mercury and 100% of the added mercury was recovered. The second test shows an increased level of addition to the flue gas with 8.4 µg/Nm^3 Hg^0 concentration expected. The measured concentration shows that in this case 60% of the mercury added reported as elemental Hg and again 100% of the Hg added was measured.

By reducing the ash loading on the system, it was possible to add elemental Hg to the flue gas at a level where meaningful testing could be performed to investigate the oxidation of elemental Hg in the reactor. However, even with the decreased ash loading in the ECO inlet, repeated QA/QC measurements through the inlet mercury sampling system indicated that within several days of continuous use, oxidation of elemental mercury was observed to occur across the QSIS filter. Figure 17 illustrates QA/QC data obtained when the QSIS filter was in a clean and uncontaminated condition.

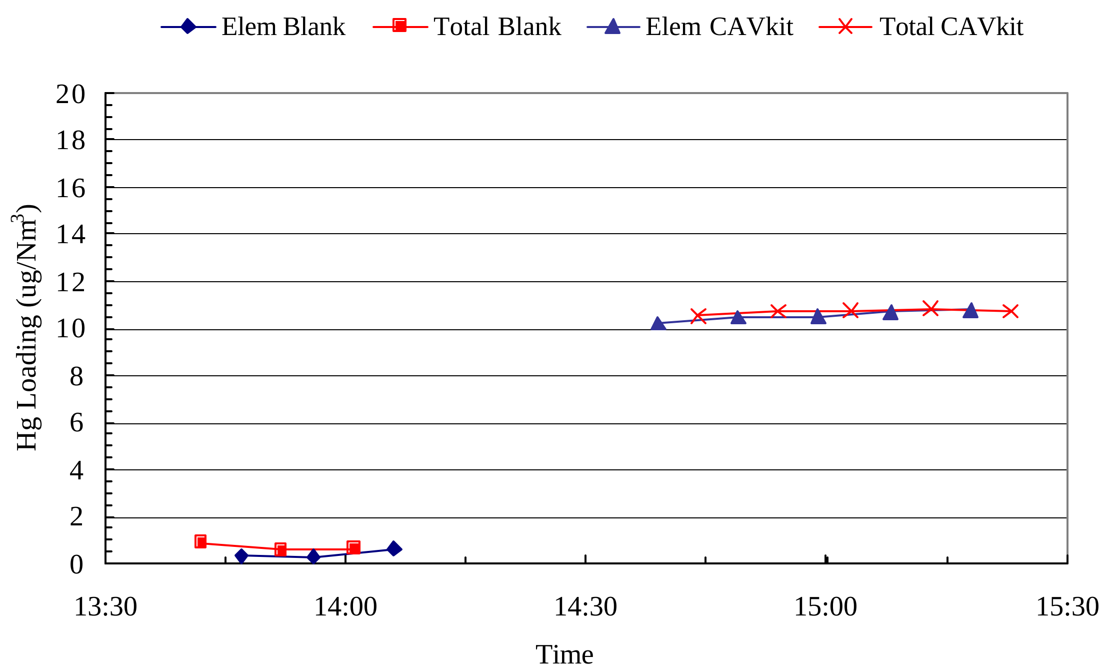


Figure 17: Results of inlet blanks and CAVkits run on a clean QSIS filter.

An analysis indicates that only 1.6% of the injected elemental mercury was lost across the entire sampling system, including the QSIS filter. After using the filter for approximately 80 hours to sample flue gas at the ECO inlet location, the same QA/QC protocols were run. The results in Figure 18 show a 25 % bias towards oxidized mercury, even though only elemental mercury was injected upstream of the filter.

In an effort to reduce the oxidation of elemental mercury across the QSIS filter, personnel at Apogee Scientific suggested a series of protocols for cleaning the filter. These protocols, discussed in a separate task report, were initially successful in keeping the oxidation of elemental mercury across this filter to a minimum. However, after operation of the QSIS probe over a number of weeks, the cleaning protocols became permanently ineffective. After this point, unacceptable amounts of elemental mercury oxidation across the QSIS filter were always observed to occur. These amounts varied from 25% to as much as 60% of the injected elemental mercury vapor. The inability to adequately clean the QSIS filter after prolonged used was observed for two filter elements and is considered to be a likely performance characteristic of these filters.

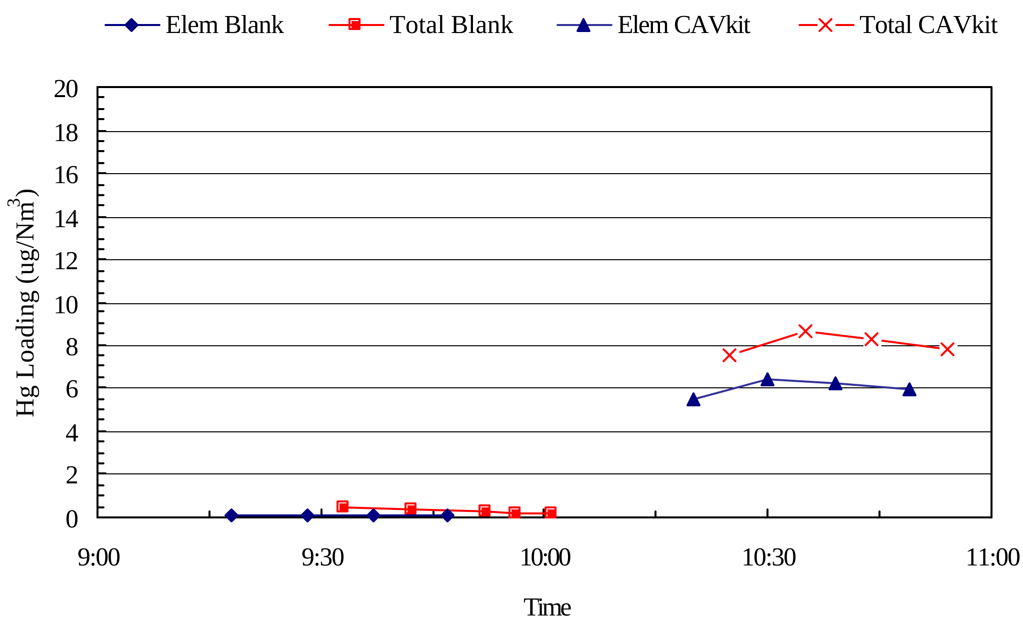


Figure 18: Results of blanks and CAVkit runs on a QSIS filter after ~80 hours of use

5.1.5 Mercury Measurements and Sampling

Measuring changes in elemental mercury concentration across the reactor were hampered by the reactive ash described above as well as by instrumentation issues described in detail in a separate technical report. Briefly, sample extraction, ash contamination, and hardware failures allowed the instruments to run for only short periods of time before maintenance was required. In addition, the inlet sampling system would not obtain quality data for a long enough period to be able to complete a series of tests. Although it would have been ideal to take measurements directly on the outlet of the reactor, the sample conditioning required to remove the acid mists produced by the reactor to avoid damaging equipment was prohibitive.

Therefore, all measurements to verify reactor performance were made using the sample probe on the ECO system outlet, which is after removal of ash by the pilot's wet electrostatic precipitator. The elemental mercury concentration was monitored while the reactor power was cycled on and off at the desired energy density. More specifically, the reactor was run for one to two hours while mercury measurements were made. After several cycles of elemental and total mercury measurements, the reactor was shut off to obtain elemental and total mercury concentrations. Without the reactor on, there is no mechanism to remove elemental mercury from the process and therefore the elemental mercury concentration at the outlet of the system is representative of the inlet mercury levels once corrected for dilution and air in-leakage. Sampling on the outlet of the system eliminates oxidation of mercury by reactive ash on the sample filter while still providing the information required to evaluate the ability of the DBD to oxidize elemental mercury. The results of the reactor testing are discussed in section 6.

5.2 Mercury Measurements in ECO Liquids and Solids

Developing a method to measure mercury in both liquid solutions and in particulate was necessary to be able to track mercury throughout the ECO process. The particulate analysis was straight forward, and the Modified ASTM Method D 6414-01 was verified for our system. The ECO process fluid proved to cause problems for the EPA Method 254.1 mercury analysis. Therefore, it was necessary to develop a new digestion procedure that would not interfere with the mercury measurement by cold vapor atomic absorption. The following is a discussion of the verification of the methods used to measure both liquid phase and particulate mercury in the ECO process.

5.2.1 Particulate Mercury Analysis (Mercury in Coal Fly Ash)

The method used to measure particulate mercury is the Modified ASTM Method D 6414-01, "Standard Test Method for Total Mercury in Coal and Coal Combustion Residues by Acid Extraction or Wet Oxidation/Cold Vapor Atomic Absorption". To verify the method a Standard Reference Material (SRM) 1633b, "Constituent Elements in Coal Fly Ash," was digested and analyzed.

Two samples of the SRM were digested and analyzed by cold vapor atomic absorption and compared to the certified value for mercury in the SRM. The results of the analysis are listed below in Table 2.

Sample	Concentration	Percent Recovery
SRM Hg Concentration	141 ± 19 ppb	------
SRM 1633b-1	161 ppb	114%
SRM 1633b-2	157 ppb	111%

Table 2: Results of SRM Analysis of Particulate Hg

The results of the verification show a mean (95% Confidence) of 159 ± 25 ppb. The method is a reliable way to measure the particulate mercury in the ECO process. An example of typical ash analysis from the pilot is shown in Figure 19.

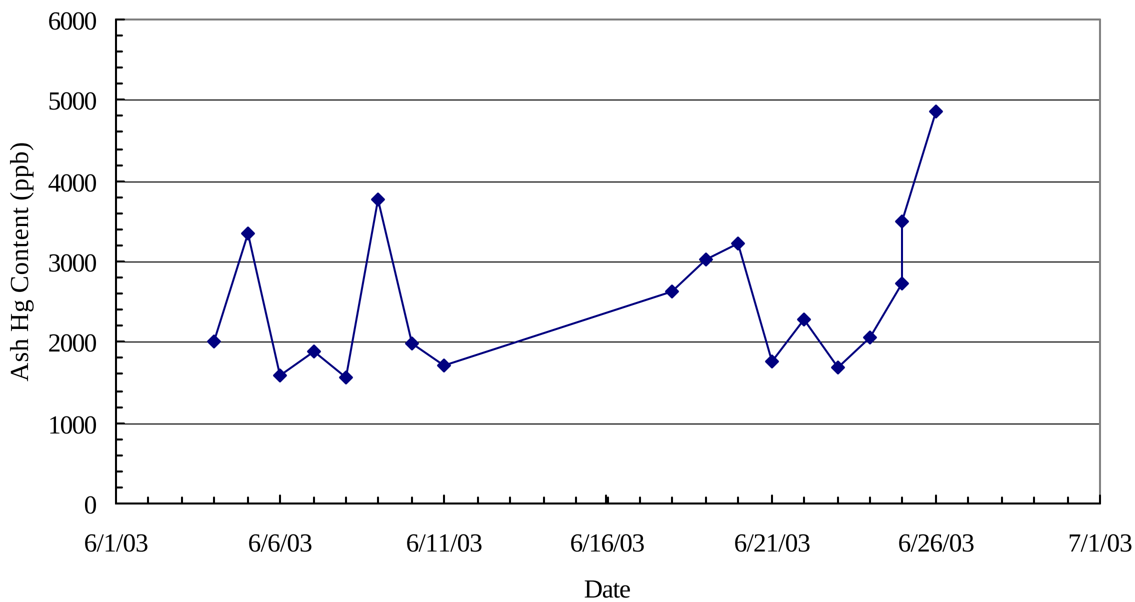


Figure 19: Typical analysis for ash Hg content

5.2.2 Mercury Analysis in Process Fluids

The Modified EPA Method 245.1, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", was modified to measure mercury in the ECO process fluids. The method was modified due to interferences created by the digestion of ECO process fluids and the subsequent analysis by cold vapor atomic absorption. Rather than a mixture of acids, only concentrated nitric acid was used to digest ECO process fluids.

Testing was done to investigate the reliability and reproducibility of the method with the modified digestion to perform mercury analysis in liquids. Duplicate samples were prepared by adding 5 mL of the test solution to 20 mL of ultra-pure concentrated (69%) nitric acid (JT Baker Ultrex Grade) in a BOD bottle. To one of the samples a known quantity of mercury standard solution (0.1 μg Hg/mL) was added. The BOD bottle was covered with foil and digested for 2 hours in a hot water bath at 90 to 95 °C. After 2 hours, it was removed from the hot water bath, cooled to room temperature, and diluted to volume with deionized water. Five milliliters of stannous chloride were added to the sample solution and it was sparged for analysis. Analysis is done using a Buck Model 400 A Mercury Analyzer. A schematic of the analysis train is shown in Figure 20.

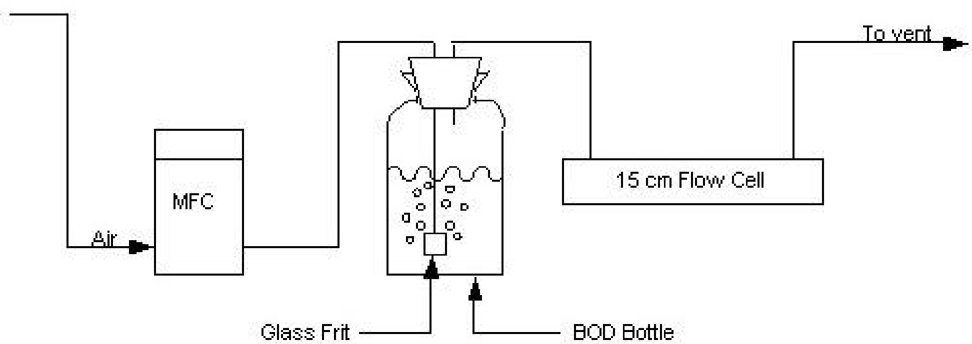


Figure 20: Analysis Train for Mercury Analysis

Testing was performed both on synthetic solutions and on process fluid samples obtained at the R.E. Burger ECO pilot. For each test, duplicate solutions were analyzed; one was analyzed unmodified and the second was spiked with a known quantity of mercury. The results of this testing are shown in

Table 3. The test shows that the digestion process developed for the ECO process fluids does not interfere with the measurement of mercury by cold vapor atomic absorption. The test solution compositions are proprietary, however solution 1 contains only the component from the ECO process fluid that required the modification of the EPA method 245.1 digestion. Solution 2 contains the 'problem' component as well as the other major components in the process fluids.

	Expected Hg Concentration (mg)	Analyzed Hg Concentration (mg)	Percent Error
Solution 1-1a	0.00	0.00	0
Solution 1-1b	0.40	0.27	-32.3
Solution 1-2a	0.00	0.00	N/A
Solution 1-2b	0.15	0.20	-22.8
Solution 2-1a	0.00	0.00	0
Solution 2-1b	0.40	0.46	14.1
Solution 2-2a	0.00	0.03	N/A
Solution 2-2b	0.40	0.47	16.9
Burger Plant Solution-1	N/A	0.21	N/A
Burger Plant Solution-2 (Spike)	N/A	0.375 (Spiked with 0.15 µg Hg) Delta = 0.375-0.211 = 0.164 µg	9.3

Table 3: Results of Digestion Verification

It has been concluded from the data that the results are consistent with error values obtained in EPA Method 245.1. At a known concentration of 0.41 µg of mercury, the method reports a standard deviation of 0.112 µg of mercury, which corresponds to a range of 0.522 µg to 0.298 µg and an error of ± 27.3%. At a know concentration of 0.06 µg of mercury the method reports a standard deviation of 0.039 µg of mercury, which corresponds to a range of 0.099 to 0.021 µg and a resulting error of ± 65%.

5.2.3 Instrument Precision Testing

Analysis on five identically prepared samples was done to test the precision of the Buck Analyzer. The samples were prepared by adding the same quantity of mercury standard to 20 mL of digestion solution in a BOD bottle. An aliquot of Solution 2 was then added to each of the bottles. The bottles were digested, diluted to volume and analyzed for mercury content. The results of the test are shown in

Table 4. The statistical analysis shows a standard deviation of 0.0006 µg Hg, with an average value of 0.057 µg. The percent relative standard deviation is 1.1 % and the

mean (95% confidence) was 0.575 ± 0.007. The conclusion of this testing is that the Buck Analyzer provided reliable and reproducible measurements.

	Concentration (μg Hg)
Sample 1	0.566
Sample 2	0.580
Sample 3	0.573
Sample 4	0.580
Sample 5	0.575

Table 4: Results from Instrument Precision Testing

5.2.4 Instrument Linearity Testing

The final verification done of the mercury measurements for the ECO process was a linearity check of the instrument. Seven solutions were prepared for analysis by adding known amounts of mercury to them. The solutions were digested by the method verified above and analyzed. The results are shown in

Table 5 and Figure 21. The figure includes the 95% confidence intervals for the measurements. This test clearly shows the Buck Analyzer responds linearly from 0.04 to 0.6 μg of mercury.

Solution	Mercury Mass (μg)	Absorbance
1	0.04	0.009
2	0.06	0.015
3	0.08	0.022
4	0.15	0.038
5	0.25	0.063
6	0.40	0.102
7	0.60	0.149

Table 5: Results from Instrument Linearity Check

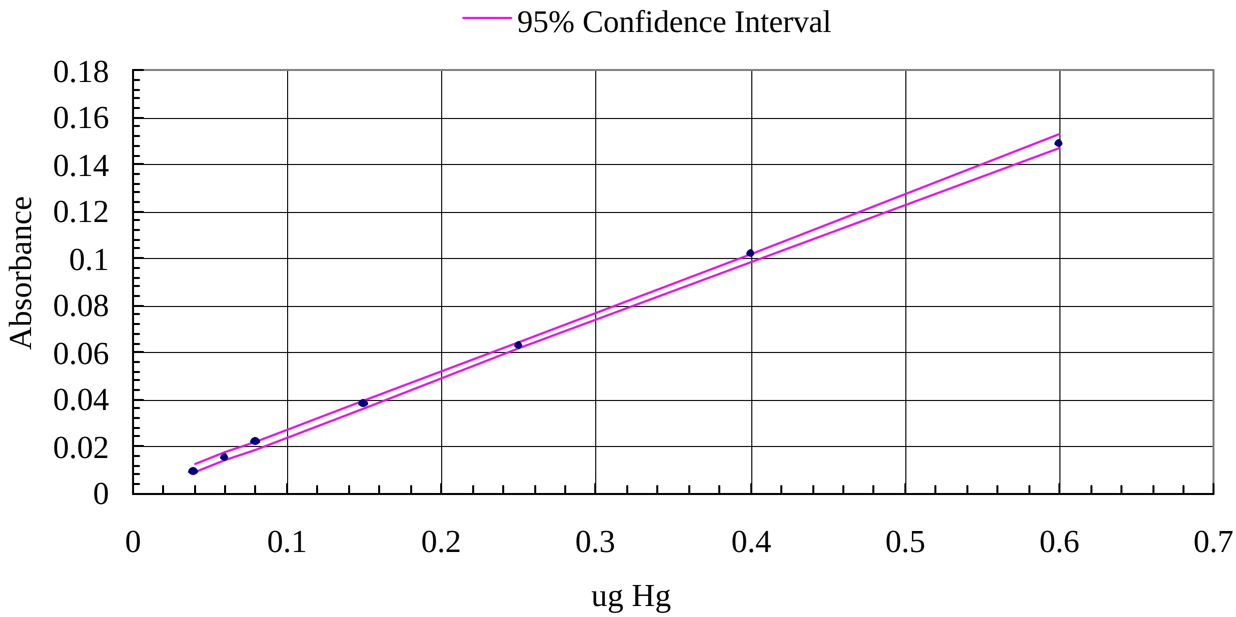


Figure 21: Mercury Analyzer Linearity Check with 95% Confidence Intervals. Statistical Analysis: $R^2 = 0.9994$, y- intercept = 0.0006, and slope = 0.2494

5.3 Contractor Testing

Validation of the Hg SCEMS instrumentation was an important part of this cooperative agreement. To do this, an outside testing service, Air Compliance Testing, was brought in to do Ontario-Hydro Testing. The complete test results and a detailed discussion can be found in a separate report however the results are summarized below.

Air Compliance Testing Inc. (ACT) conducted three days of baseline validation testing of the installed Hg SCEMs systems. The validation testing involved twenty-three hours of testing using the Ontario-Hydro Method during May 8-10, 2002. During testing, the two PS Analytical Hg SCEM systems were operated using Baldwin Environmental sample probes at the system inlet and outlet. The instruments were alternated between Elemental Hg and Total Hg measurements every five minutes. A blank was obtained prior to the testing event to allow the measurements to be blank subtracted and averaged over the time period of the ACT sample collection. Calculations of oxidized mercury levels were made by subtracting the PSA elemental Hg levels from the PSA total Hg levels.

For comparison, ACT collected three samples at the system inlet consisting of one three-hour run and two four-hour runs and three samples at the system outlet each consisting of four hours runs. A comparison of the data obtained by the PS Analytical instruments and ACT is provided in Table 6 below. The PS Analytical total and elemental Hg measurements were subtracted to give the oxidized Hg concentration which is reported below in Table 6.

	PSA Elemental µg / dscm	ACT Elemental µg / dscm	ACT Hg^0 Detection Limit	PSA Oxidized µg / dscm	ACT Oxidized µg / dscm	ACT Hg^{2+} Detection Limit
Inlet Run 1	0.53 ± 0.10	<0.52	0.52	3.96 ± 0.68	5.28	0.22
Inlet Run 2	0.06 ± 0.03	<0.35	0.35	4.68 ± 0.75	5.70	0.26
Inlet Run 3	0.19 ± 0.07	<0.42	0.42	5.74 ± 0.75	6.46	0.27
Outlet Run 1	0.58 ± 0.10	0.58	0.48	0.15 ± 0.04	<0.37	0.37
Outlet Run 2	0.40 ± 0.05	0.58	0.38	0.13 ± 0.04	<0.32	0.32
Outlet Run 3	0.45 ± 0.03	1.09	0.37	0.15 ± 0.05	<0.31	0.31

Table 6: Comparison of Air Compliance Testing and Powerspan Hg SCEMS results.

On the inlet, the elemental Hg measured by ACT was below the detection limit (BDL) of the method and the PS Analytical instruments reported 0.53, 0.06, and 0.19 $\mu g/Nm^3$, which confirm the measurements were BDL. For oxidized Hg, ACT measured 5.28, 5.70, and 6.46 $\mu g/Nm^3$ compared to 3.96, 4.68, and 5.74 $\mu g/Nm^3$ measured by the PS Analytical instruments in Inlet Run 1, 2, and 3, respectively. The oxidized Hg concentration increased for both ACT and the PS Analytical instruments from Run 1 to Run 3. The average error in the PS Analytical instruments compared to the ACT measurements was 18% for the oxidized Hg measurement on the inlet.

On the outlet, the elemental Hg measured by ACT was 0.58, 0.58, and 1.09 $\mu g/Nm^3$ compared to 0.58, 0.40, and 0.45 $\mu g/Nm^3$ measured by the PS Analytical instruments in Outlet Run 1, 2, and 3, respectively. The outlet oxidized Hg measured by ACT was BDL and measured by the PS Analytical instruments was 0.15, 0.13, and 0.15 $\mu g/Nm^3$ for Outlet Run 1,2, and 3 respectively. The average error in the PS Analytical instruments compared to the ACT measurements was 29% for the elemental

Hg measurements on the outlet. However, if Run 3 is considered an outlier due to the large increase in elemental Hg seen, the average error decreases to 11%.

This testing data suggests a reasonable agreement between the Ontario-Hydro method test results and the Hg SCEMS test results. The complete results for the ACT test event are included in the Task 1 Technical Report, but a brief summary of performance is shown in Table 7.

Hg Fraction	**ECO Inlet**	**ECO Outlet**	**Removal**
Particle Bound Hg (µg/dscm)	0.62	0.016	97.4 %
Oxidized Hg (µg/dscm)	5.81	0.022	99.6 %
Elemental Hg (µg/dscm)	0.16	0.75	
Total Hg (µg/dscm)	6.59	0.79	88.0 %

Table 7: Summary of Ontario-Hydro Test

6.0 Results and Discussion

Although testing under this cooperative agreement was focused on mercury removal from the flue gas of coal fired boilers, the ECO process is designed to be a multi-pollutant control technology. For the technology to be successful, it must remove high levels of NO_x, SO_2, and particulate matter (PM), in addition to the Hg removal. The discussion below focuses on the overall performance of the ECO Process during operation of the Burger Pilot Unit.

6.1 Removal of NO_x and SO_2,

Presented in Figure 22 and Figure 23 is pilot data showing NO_x and SO_2 concentrations and removal levels measured over 12 hours of operation. Figure 22 shows that NO_x removal over the test period averaged 90% with an inlet level of 250 ppm. Figure 23 shows 98% SO_2 removal from an average inlet concentration of 1320 ppm. Also presented in Figure 23 is the outlet ammonia concentration (shown in parts per billion), which averaged 1.0 ppm.

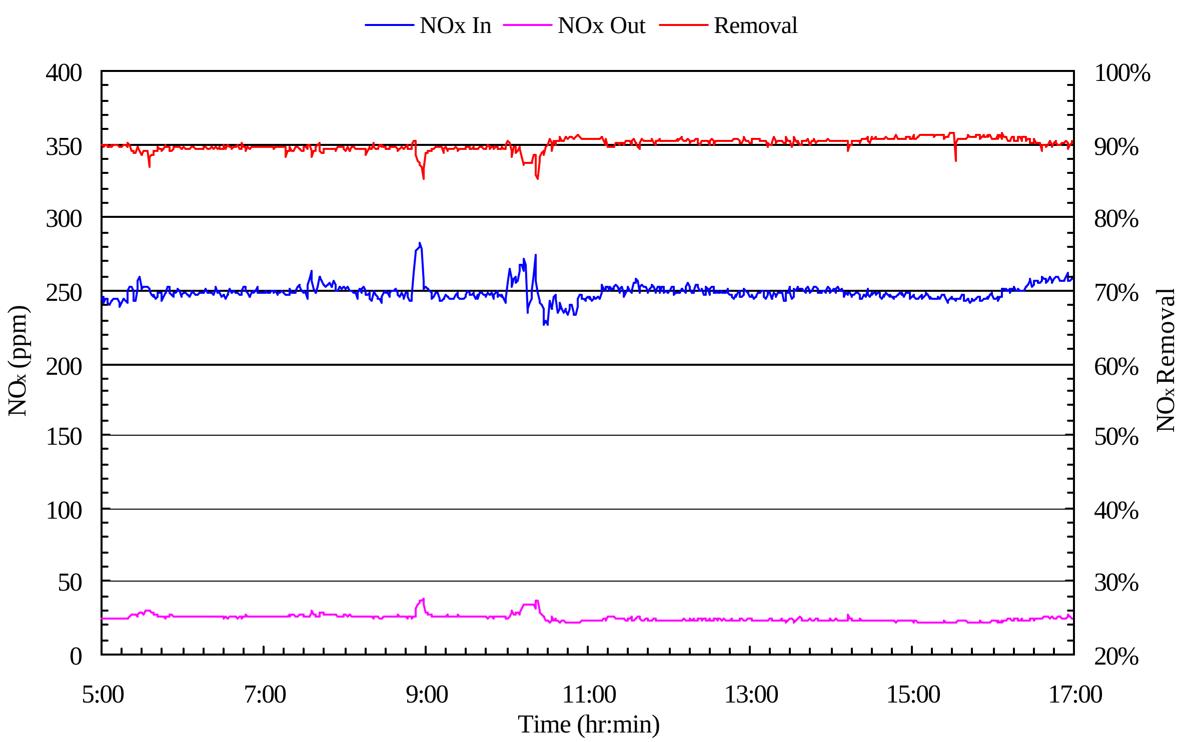


Figure 22 NO_x performance data from the Burger pilot unit

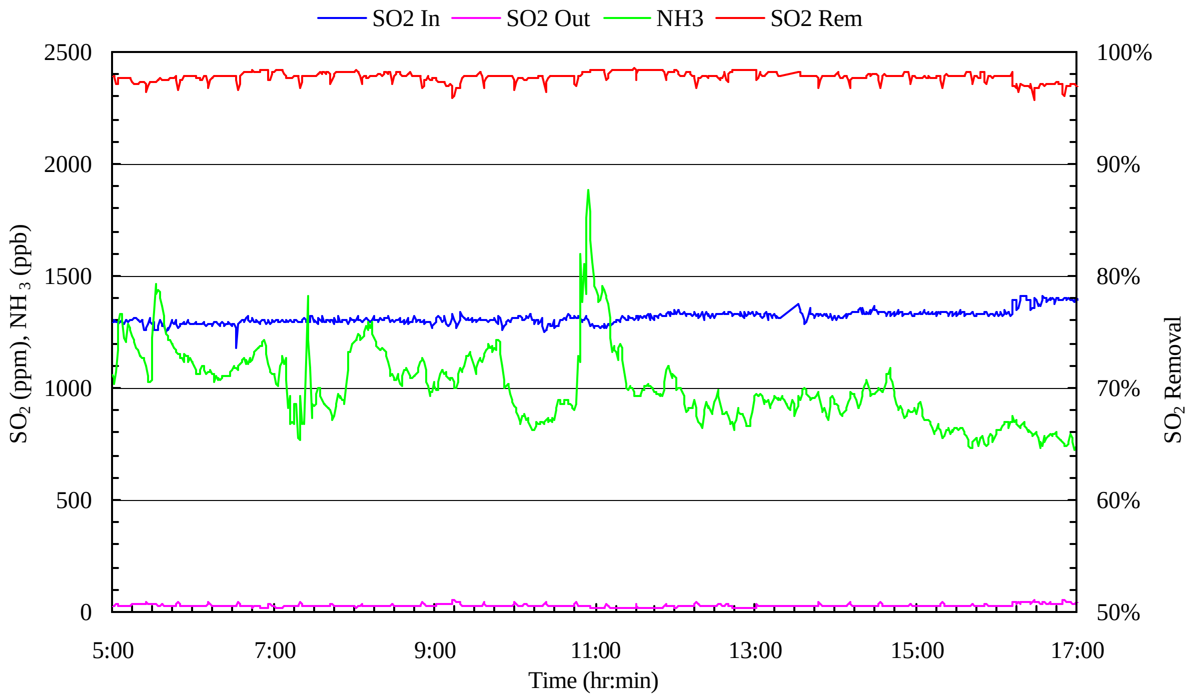


Figure 23 SO_2 performance data from the Burger pilot unit

The removal levels and concentrations shown in the figures are typical of the performance measured during normal operation of ECO process.

6.2 Mercury Removal in the ECO Process

There were several levels of testing done looking at mercury removal in the ECO process at the Burger pilot. They include (i) overall removal across the system with native and elevated mercury concentrations, and (ii) oxidation of elemental mercury by the DBD reactor. These are discussed in more detail below.

6.2.1 Total Hg and PM Removal

Mercury test data from the pilot's installed Hg SCEM over a 12 hour period is presented in Figure 24. It shows 90% Hg removal from an inlet concentration of approximately 4 µg/Nm^3. An elemental Hg addition system was installed at the ECO pilot to increase the concentration of elemental Hg in the flue gas stream.

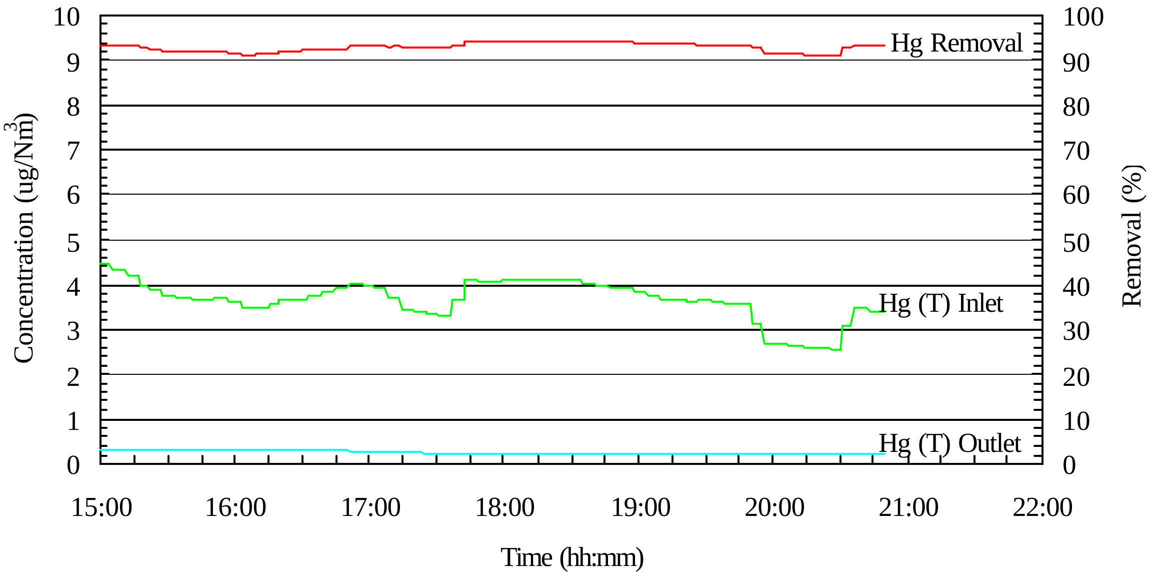


Figure 24: Hg Concentrations and Removal of Native Hg at the ECO Pilot

This was done in order to demonstrate the ability of the ECO process to oxidize and capture elemental Hg in actual flue gas, as had been shown using synthetic flue gas in the laboratory. Because of the high ash content in the flue gas stream at the inlet to the ECO process, the elemental Hg that was added was measured as oxidized Hg. However, Figure 25 shows that high levels of Hg removal were also achieved at the elevated oxidized Hg concentrations.

The recent Ontario-Hydro test results obtained after addition of the ammonia scrubber in early 2002 show total Hg removal levels consistent with those measured at the ECO pilot prior to the scrubber installation. Method 29 measurements were made in 2000 by Air Compliance Testing during which the total mercury removal level was measured to be 81.6% [13]. During those same tests the removal of arsenic, barium, chromium, copper, lead, manganese, nickel and phosphorous exceeded 99%. Air Compliance Testing also made particulate matter measurements in 2000 [14]. The results showed that 99.6% of the total particulate matter was captured in the ECO process. The testing also measured 96.7% capture of particles less than 3 microns in size.

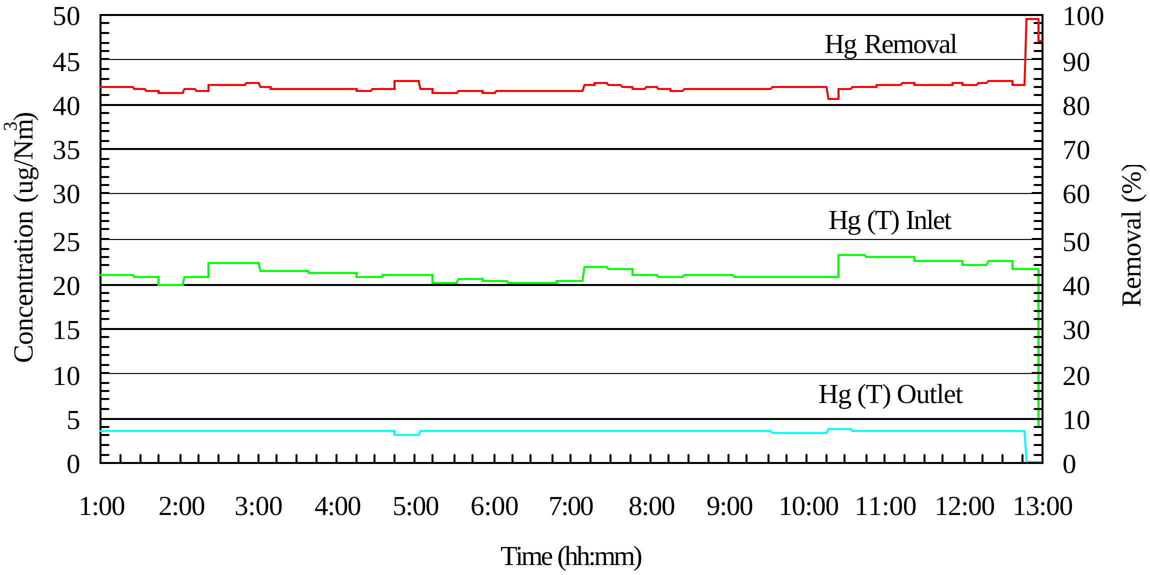


Figure 25: Hg Concentrations and Removal of Elevated Hg at the ECO Pilot

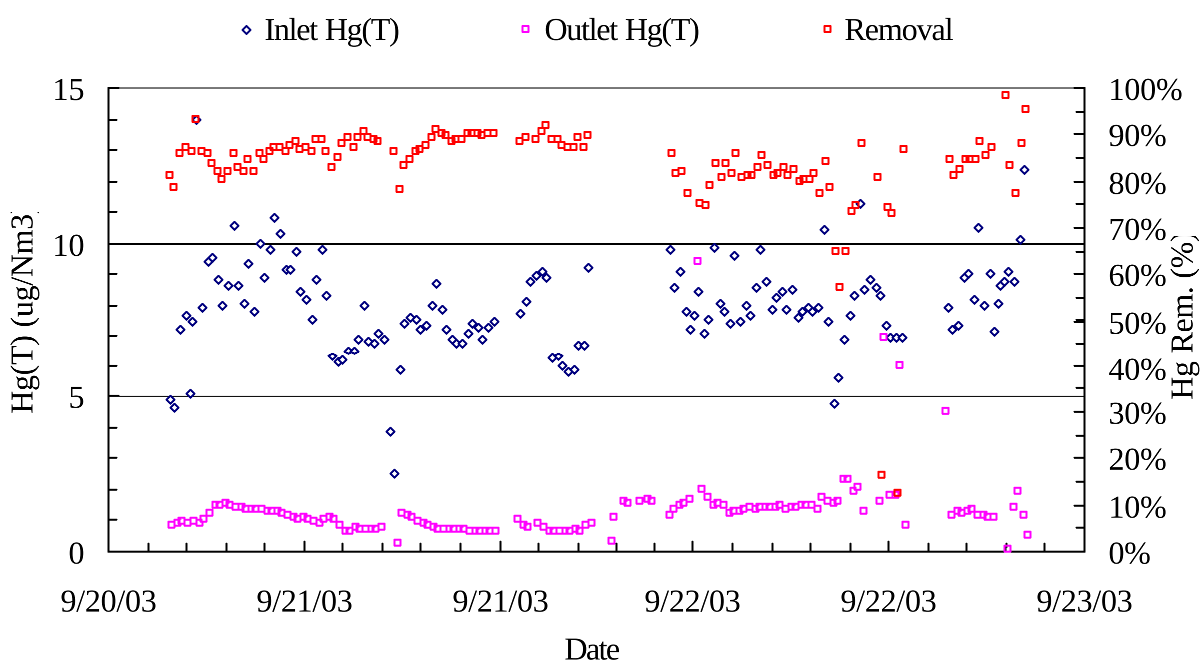


Figure 26: Hg data for extended run

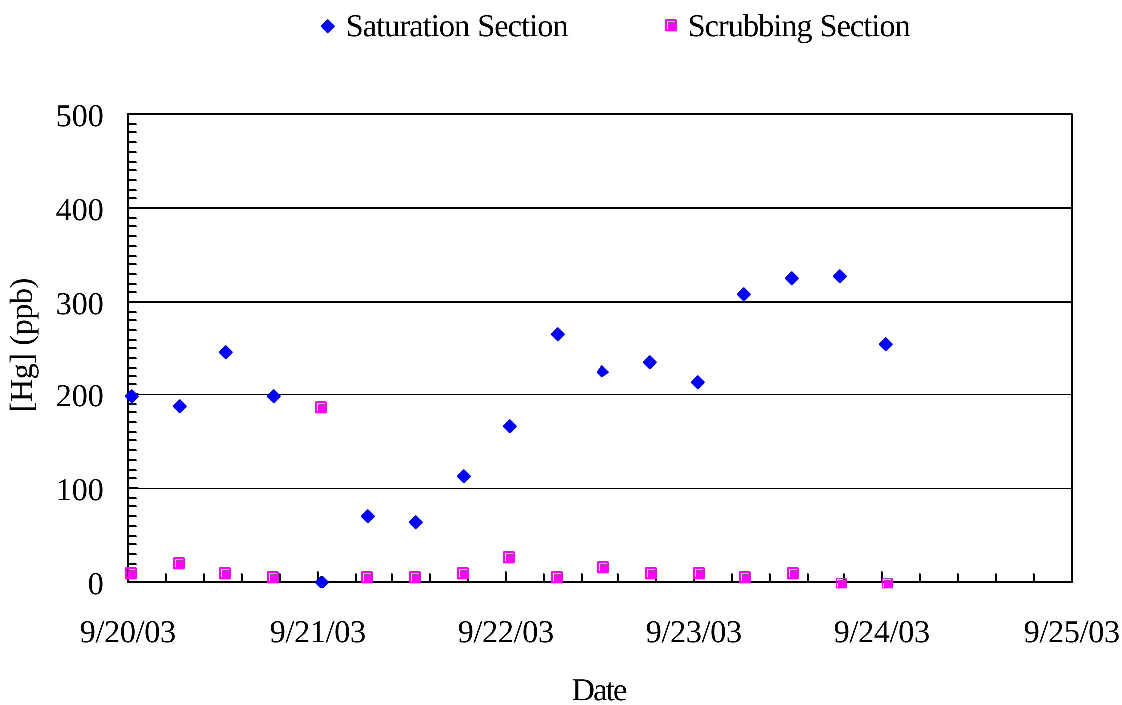


Figure 27: Liquid analysis corresponding to extended run

It was also possible to get one extended run with mercury measurements spanning several days. The mercury removal data is shown in Figure 26 with the corresponding liquid analysis in Figure 27.

6.2.2 Mercury Oxidation in DBD Reactor

Testing of the barrier discharge reactor's ability to oxidize elemental mercury was performed using two flue gas configurations. The first configuration was one in which flue gas was supplied to the ECO pilot from the inlet to the Burger plant's ESP and utilized the pilot's cyclone separator and dry ESP for ash removal. In this configuration, the ash loading on the ECO reactor was measured to be 0.13 gr/dscf. In the second configuration, after installation of the new ductwork, flue gas was supplied from the outlet of the Burger plant's ESP, substantially reducing the ash loading on the ECO pilot. Measurements of the ash loading at the outlet of the Burger plant ESP showed an average ash loading of 0.009 gr/dscf.

Testing of elemental mercury oxidation by the barrier discharge reactor was performed using elemental mercury injection into the inlet flue gas duct and mercury measurements at the outlet of the ECO system only. Inlet elemental mercury measurements could not be reliably made due to the sampling difficulties encountered as a result of ash contamination of the sample system at the inlet to the pilot. Sampling efforts at the ECO pilot inlet are discussed briefly in this report (section 5.1) and in more detail in a separate task report.

Shown in Figure 28 are results from testing where the DBD reactor was operated at a power level of 18 w/scfm and the flue gas flow through the ECO pilot was held at 1500 scfm with an inlet elemental Hg concentration determined to be ~2.5 ug/Nm^3. The inlet elemental mercury concentration was taken to be that measured at the outlet of the system with the DBD reactor secured. Measurements at the outlet with and without the DBD reactor operating show the oxidation of elemental Hg by operation of the DBD.

At the completion of these tests, the amount of elemental mercury injected into the ECO inlet duct was increased and the reactor on/off sequence was performed again. The data from these tests are presented below in Figure 29.

The results of elemental mercury oxidation by the barrier discharge reactor in the high reactor inlet ash loading configuration are summarized above in Table 8. The reported inlet elemental Hg concentration is the average concentration measured at the outlet of the system with the DBD reactor secured.

The reactor testing was repeated after installation of a new duct run, connecting the ECO pilot to the Burger Plant dry ESP outlet. This configuration is referred to as "normal ash loading" because it represents ash loading expected in a commercial ECO installation. Installation of the new duct was intended to alleviate the problems encountered with injection and measurement of elemental Hg at the inlet to the ECO pilot. Although much reduced, some oxidation of elemental Hg continued to take place in the sampling system and inlet ductwork. Therefore testing was conducted as in the initial duct configuration where the inlet concentration was taken to be that measured at the outlet of the pilot with the DBD reactor secured.

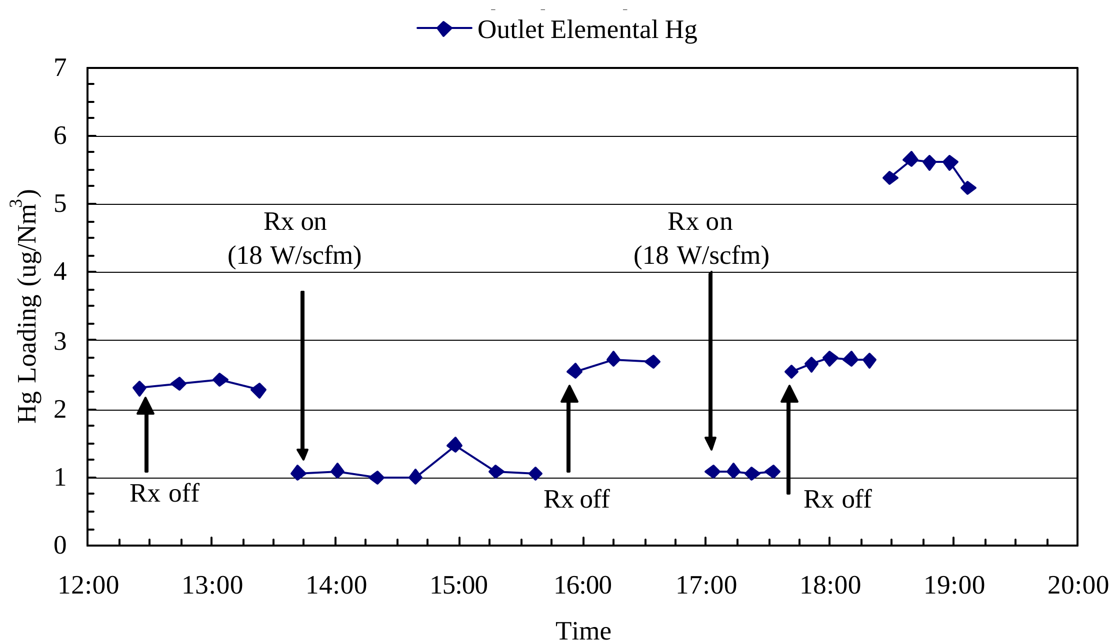


Figure 28: ECO DBD reactor performance in high ash loading environment

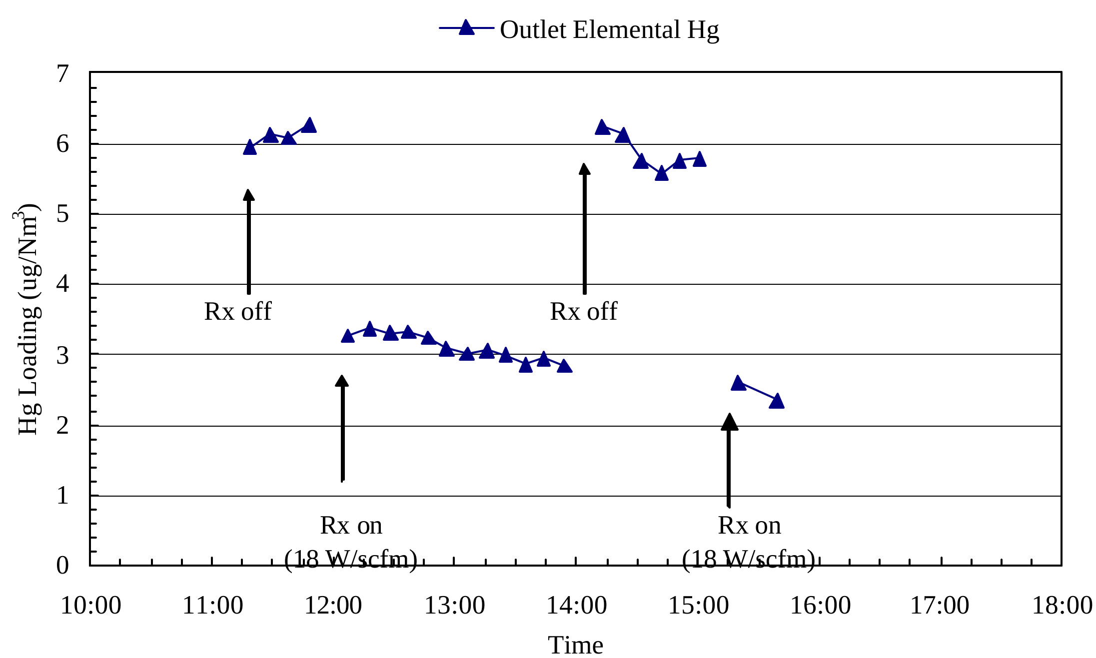


Figure 29: Conversion of elemental mercury at 18 watts/scfm at higher concentration with high ash loading

The tests were conducted at two flowrates: 2000 scfm and 2500 scfm. During each of these runs the DBD reactor was cycled between 0, 15, 20 and 25 Watts/scfm. The results from the low flow tests are presented below in Figure 30. Based on calibration of the mercury addition system, the concentration of elemental mercury injected into the duct during these tests was 8.7 $\mu g/Nm^3$.

Run	DBD Reactor Inlet Elemental Hg ($\mu g/Nm^3$)	DBD Reactor Outlet Elemental Hg ($\mu g/Nm^3$)	Elemental Hg Conversion.
1	2.3	1.1	53%
2	2.7	1.1	60%
3	6.1	3.1	49%
4	5.9	2.5	58%

Table 8: Elemental Hg conversion at 18 W/scfm, 1500 scfm gas flow

The results from the high flow (2500 scfm) tests are presented below in Figure 31. The result of increasing the reactor power density is also visible in the data. We calculated that the amount of elemental mercury injected into the duct during the high flow tests was 7.5 $\mu g/Nm^3$. The results of both high and low flow test as a function of reactor power are summarized below in Table 8. A plot of elemental Hg oxidation as a function of reactor power density, for both the high and low flow tests is presented below in Figure 32. The figure shows an increase in elemental Hg oxidation as a result of increasing DBD reactor power.

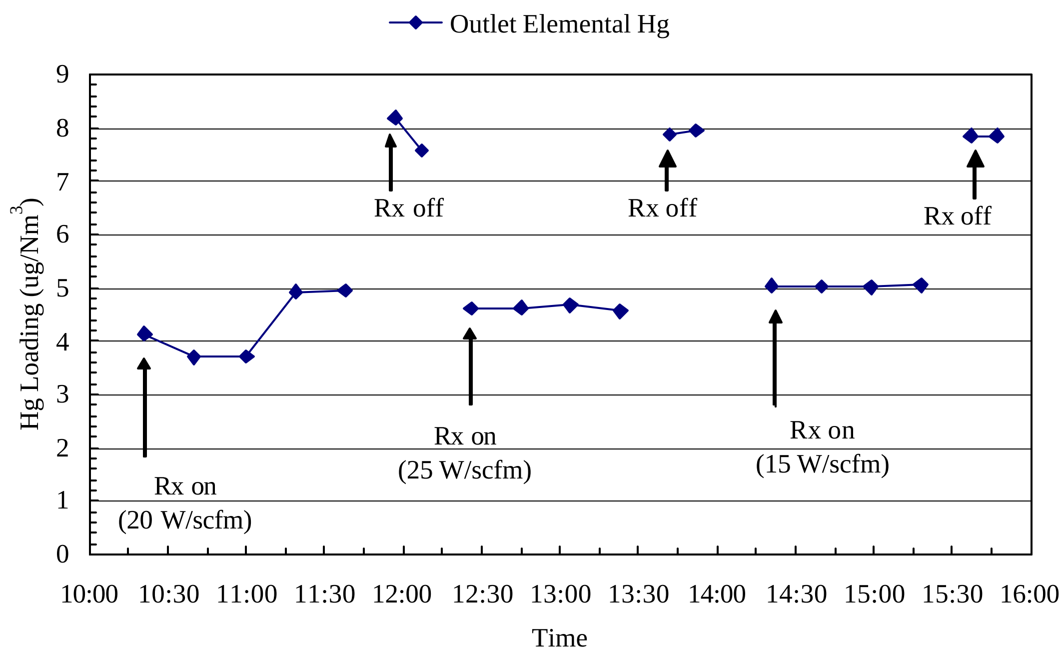


Figure 30: Elemental mercury conversion at 20, 25 and 15 W/scfm with normal ash loading at a flow rate of 2000 scfm, Runs 1,2 and 3

Run	Rx Power Density (W/scfm)	Flue Gas Flow (scfm)	Elemental Hg Conversion
1	15	2000	36%
2	20	2000	46%
3	25	2000	42%
4	15	2500	57%
5	20	2500	64%
6	25	2500	75%

Table 9 Elemental Hg conversion at 15, 20 and 25 W/scfm at two different flue gas flow rates.

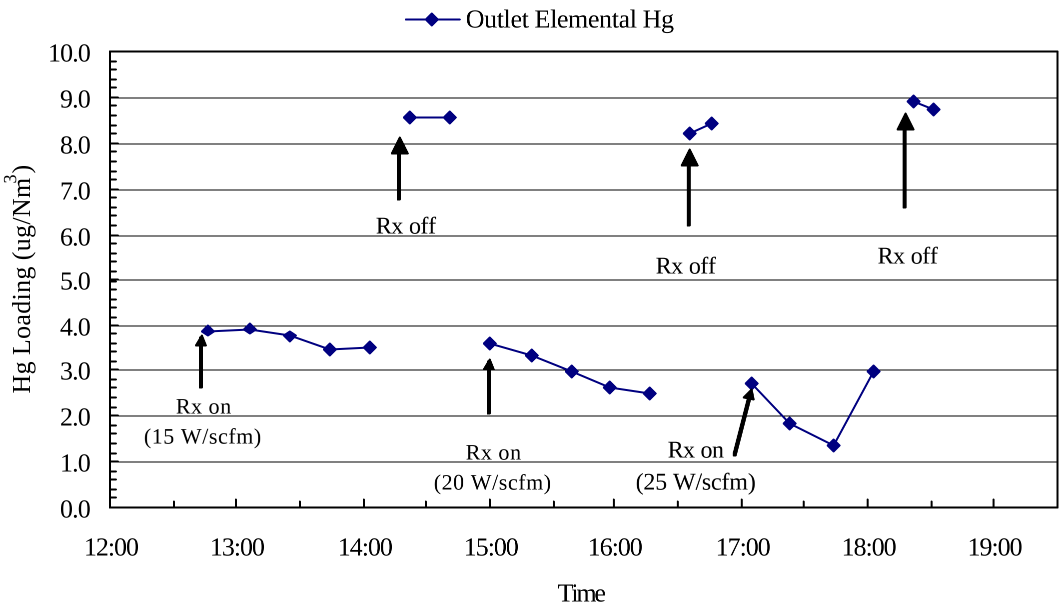


Figure 31: Elemental mercury conversion at 15, 20 and 25 W/scfm with normal ash loading at a flow rate of 2500 scfm, Runs 4,5 and 6.

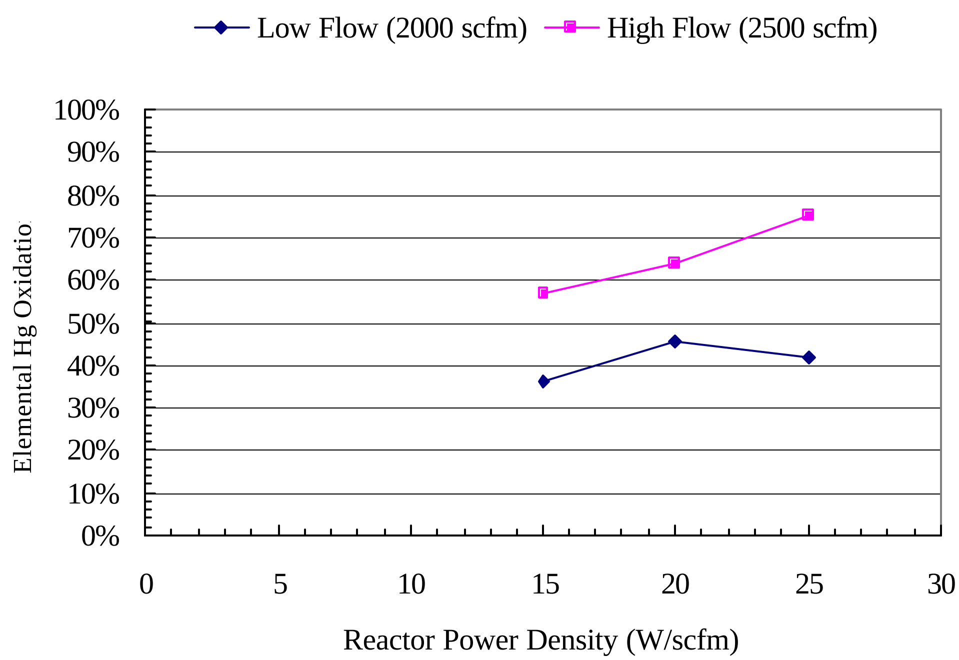


Figure 32: Elemental Hg conversion at 15, 20 and 25 W/scfm at two different flue gas flow rates.

6.3 Mercury Removal from Liquid Co-Product

Two separate adsorbents were investigated for mercury removal from ECO Process fluid; KeyleX® (Figure 33) and Mersorb® LW (Figure 34). KeyleX is a sulfonated chelating ion exchange resin that is capable of removing oxidized Hg through

Figure 33: KeyleX cartridge and cartridge holder

Figure 34: Picture of Mersorb LW for mercury removal in ECO Process Fluid

adsorption sold and supported by SolmeteX. It has been used in commercially to decrease mercury in waste incinerator wastewater at flow rates of up to 30 gpm. The absorption capacity of the resin is 10 wt% Hg. For example, 1 cubic foot of resin weighs 45 pounds and can capture 4.5 pounds of mercury. [16]

Mersorb LW, manufactured by Nucon International, is a sulfur impregnated activated carbon. The high surface area of the activated carbon (1000 m^2/g by N_2 BET test method) and affinity of Hg for elemental sulfur creates an adsorbent that can hold a maximum of 75 wt % Hg. For example, 1 cubic foot of Mersorb weighs 34 pounds and can capture a maximum of 25.5 pounds of mercury. [17]

A flow diagram of the mercury removal system is shown in Figure 35. The liquid is passed through a 0.5 micron filter and a guard column filled with standard activated carbon (Nusorb) before being treated with Mersorb. The Nusorb absorbs hydrocarbons in the effluent solution, extending the life of the Mersorb. There is one Nusorb section which is 2" CPVC pipe and is 18" long and two Mersorb LW sections also made with 2" CPVC pipe that are 36" long each. The system flow rate is 250 cc/min giving a residence time in each Mersorb LW bed of 7.5 minutes for a total of 15 minutes. Samples are taken and analyzed for mercury after each processing step. Ammonia can be added to the effluent stream to adjust the pH as necessary.

The laboratory test bed, shown in

Figure 36, could use either Mersorb or KeyleX as the absorbent by replacing the Nusorb and Mersorb canisters with the KeyleX cartridge shown in Figure 33. The same flowrate was used through the KeyleX system

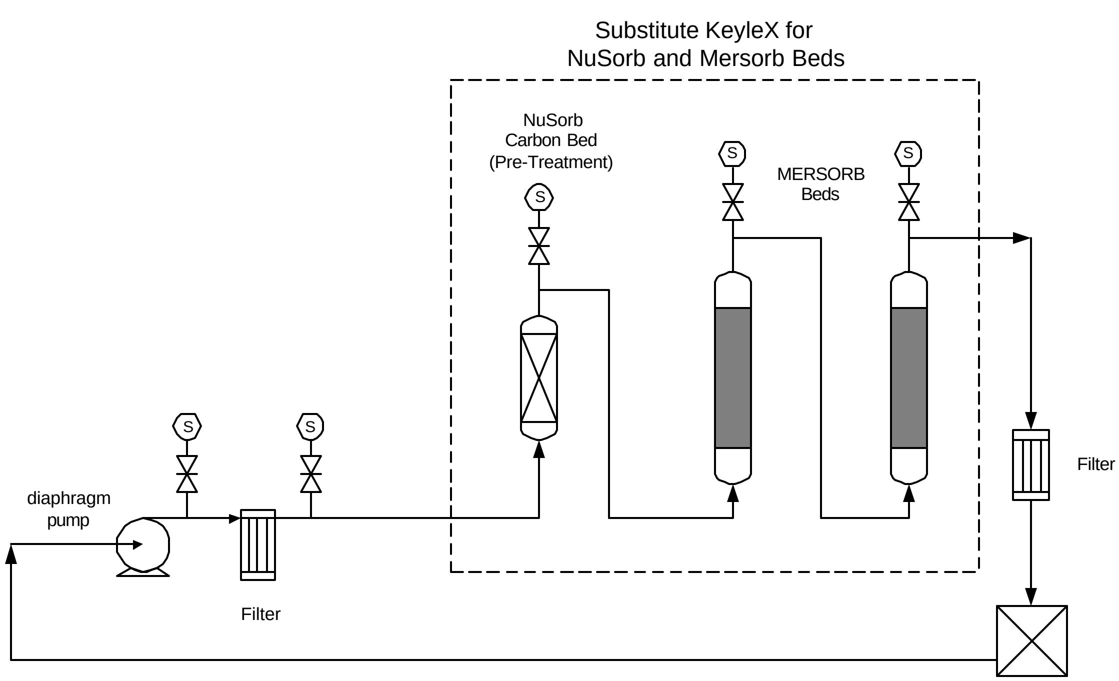
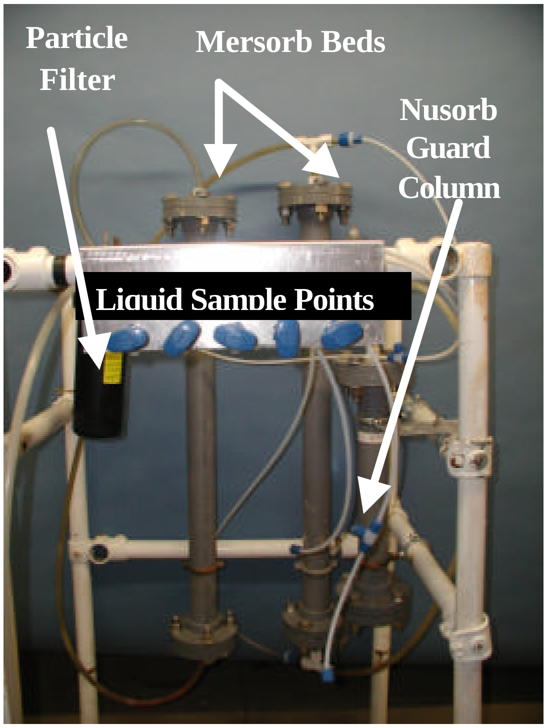


Figure 35: Flow diagram of Mercury Removal Absorption Bed



(a) (b)

Figure 36: Picture of (a) laboratory Mersorb Test Bed and (b) Pilot Mersorb Bed

giving a residence time of 2.3 minutes. Parametric testing of pH and liquid composition was done both the Mersorb and KeyleX to determine the mercury removal efficiency of the adsorbents. The results are shown in Table 10. It is clear that the smaller KeyleX bed can remove mercury as well as the Mersorb LW bed with a smaller system. However, it

KeyleX			Mersorb LW		
PH	Initial Hg Concentration (ppb)	Final Hg Concentration (ppb)	pH	Initial Hg Concentration (ppb)	Final Hg Concentration (ppb)
4	400	BDL	4	431	12
5	385	BDL	5	276	10
6	305	50	6	454	17

Table 10: Mercury removal performance for KeyleX and Mersorb LW

is necessary to do further testing to determine the capacity and overall cost of the two systems for commercial operation. Only the Mersorb LW has been tested at the pilot scale as discussed below.

The mercury removal bed at the Burger pilot had the same process flow as the laboratory system. The Mersorb canisters were made of 10” PVC pipe 36” long and the Nusorb canister was 24” long. Again, particle filters were used to remove particles larger than 0.5 microns. The flowrate through the system was typically 0.5 gpm providing a residence time of 15 minutes. Like the laboratory system, ammonia addition to the stream was automated to adjust pH as necessary.

Using Mersorb as an adsorbent, mercury levels in the pilot effluent liquid have been reduced to less than the limit of detection (~20 ppb) from concentrations as high as 200 ppb. Subsequent crystallization of the effluent produced ammonium sulfate crystals contained 21% nitrogen, as would be expected for ammonium sulfate. Figure 37 shows photographs of the crystals obtained during the crystallization process. Metals analysis [15] confirmed that the mercury concentration in the crystal product was below detectable limits.

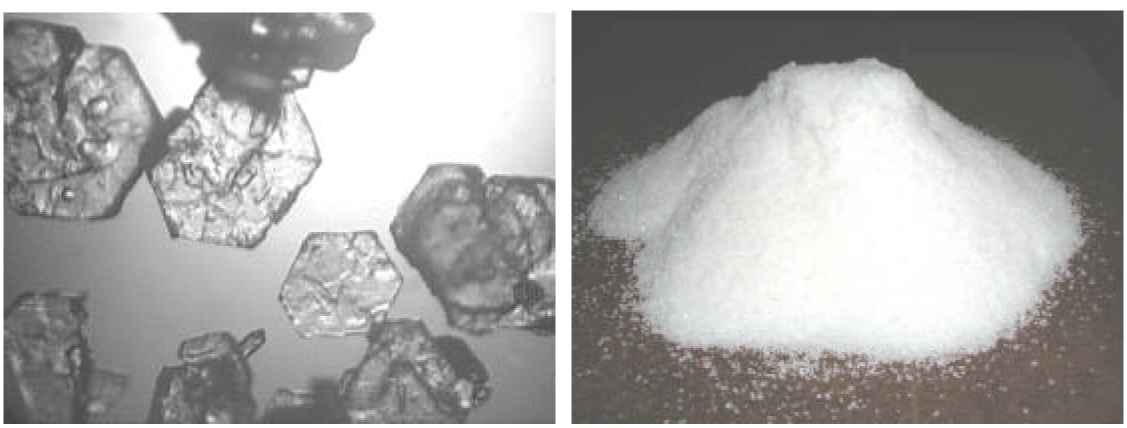


Figure 37: Ammonium Sulfate Crystals produced from effluent of R.E. Burger Pilot

7.0 Conclusion

A system for sampling gas phase mercury in the flue gas of the ECO pilot at FirstEnergy's Burger Power Plant was selected, installed and operated to support testing of mercury removal in a multi-pollutant control technology. The system chosen was provided by PS Analytical and included sample probes, sample conditioners, a stream selector and an atomic florescence spectrometer. The PS Analytical equipment performed well in the clean flue gas environment at the outlet of the ECO process. However, the ash loading at the inlet of the ECO system, and the ability of the ash to oxidize elemental mercury, proved problematic for accurately determining the concentrations of elemental and oxidized mercury in the flue gas. Extensive efforts to improve the inlet flue gas measurements included testing of multiple sample filters, changing sample probe operating conditions, consulting with industry experts, installation and testing of an inertial separation based sampling system, and installation of ductwork in order to provide flue gas with a reduced ash loading to the ECO pilot . None of these efforts were successful at providing a system and conditions where routine and accurate measurement of the mercury species contained in the flue gas entering the pilot could be made.

The inability to accurately measure gas phase mercury species in the inlet gas substantially restricted the investigation of elemental mercury oxidation by the ECO process' barrier discharge reactor. In order to provide a measure of the inlet elemental mercury concentration, measurements were made at the outlet of the ECO process with the barrier discharge reactor secured. Testing in this manner did not allow sufficient time for the entire ECO process to reach a steady state, restricting the parametric investigation to operation of the barrier discharge reactor. Planned parametric testing of the ammonia scrubber and wet electrostatic precipitator could not be accomplished.

Protocols were developed throughout the testing for calibration, maintenance, troubleshooting and repair of the installed gas phase mercury monitoring system. Operating procedures were also developed, including frequent checks by the ECO pilot operators. Frequent monitoring and maintenance were found to be required in order to keep the instrumentation operating for more than a few hours at any one time. Two complete measurement systems were installed at the ECO pilot, with the ability to sample from four locations in the process. However, the extensive efforts required to keep the measurement systems operating restricted sampling to two locations at any one time.

In addition to gas phase mercury measurements, methods were adapted and tested for measurement of mercury in ECO process fluids. The measurement of mercury in captured ash was done using a standard method with modification. Mercury measurement in the ECO scrubber fluids required modification of standard methods to eliminate matrix effects in the mercury digestion process. The modified method was successfully tested and used in the project.

Using measurements at the outlet of the system only, oxidation of mercury by the dielectric barrier discharge reactor was tested over a range of energy densities and gas flow rates. The limited testing conducted has shown: (i) increasing energy density in the reactor increases the conversion of elemental mercury, (ii) the oxidation of elemental mercury is not dependant on inlet elemental mercury concentration in the range of 2 – 8 $\mu g/Nm^3$, and (iii) increasing the gas flow through the reactor increases the oxidation of elemental mercury.

Testing of the DBD reactor demonstrated that increasing the reactor power density (energy density) from 15 W/scfm to 25 W/scfm increased the elemental mercury conversion from 57% to 75% at a nominal flow rate of 2500 scfm through the reactor. Increasing the energy density in the reactor increases the concentration of oxidizing species produced by the barrier discharge process, increasing the rate of reaction (10). This behavior is consistent with that seen in the conversion of NO to NO_2 and HNO_3, where increasing energy density increases the rate of oxidation.

A test of DBD performance at different inlet mercury levels suggests that the oxidation of elemental mercury is not dependent on the inlet elemental mercury concentration. Elemental mercury concentrations of 2.5 ug/Nm^3 and 6.0 ug/Nm^3 were tested with a flowrate of 1500 scfm and an energy density of 18 watts/scfm resulted in a conversion percentage of 56%±5% and 53±6%, respectively. This result varies from the behavior exhibited by the reactor for NO_x oxidation. In the case of NO_x, the molar conversion to NO_2 and HNO_3 is constant for a given reactor energy density resulting in a higher conversion percentage as the inlet NOx concentration is reduced. For example, operation of the discharge reactor to create 100 ppm of HNO_3 from NO will require the same energy input at an inlet NOx concentration of 200 ppm (resulting in 50% NOx conversion) as at an inlet concentration of 250 ppm (40% NOx conversion). For elemental mercury oxidation, in the range that was tested, there was no decrease in conversion percentage with increased elemental mercury concentration. It is thought that at the mercury concentrations present in flue gas, the collision rate between elemental Hg and the oxidizing radicals produced by the reactor is the rate limiting step. Therefore, when the concentration of elemental mercury in the flue gas is increased, the rate of reaction and the conversion efficiency of the reactor also increase.

Parametric testing looking at the effect of velocity through the reactor on the conversion efficiency of elemental mercury showed greater conversion as the gas velocity increased. At an energy density of 15, 20 and 25 W/scfm, increasing the flue gas flow through the reactor from 2000 scfm to 2500 increased the conversion of elemental mercury from 36% to 57%, 46% to 64%, and 42% to 74% respectively. Again, this is in contrast to the mechanism for NO_x oxidation in the DBD reactor. For NO_x, oxidation and conversion efficiency are related to the concentration of NO_x and the energy density of the non-thermal plasma. For mercury, the results show that the increased flow has increased conversion efficiency. This result may be due to increased gas mixing resulting from higher gas velocities.

Pilot testing of the ECO process under this cooperative agreement has shown (i) with extensive monitoring, Hg SCEMS can be used to obtain information on mercury speciation in coal combustion produced flue gas, (ii) the ECO DBD reactor oxidizes elemental mercury in flue gas streams with 2- 10 $\mu g/Nm^3$ of elemental Hg, and (iii) the ECO process can obtain high removal levels of multiple pollutants (>80% Hg, 98% SO_2, 90% NO_x, 95% of fine particles) with a single installation, (iv) Hg captured in the ECO scrubber is removed from the scrubber effluent, allowing for economic disposal of captured Hg in a hazardous waste storage facility. Extensive parametric testing planned for the program, including testing at high inlet elemental Hg levels, could not be completed due to the difficulties encountered with establishing and measuring representative elemental Hg concentrations in the flue gas entering the ECO pilot.

The pilot testing showed ECO to be a multi-pollutant control process capable of achieving high removal levels for NO_x, SO_2, particulate matter and Hg. Successful testing has led to the installation of an ECO commercial demonstration unit at FirstEnergy's Burger Power Plant. The demonstration unit treats a 50 MWe equivalent flue gas slipstream and is intended to show the commercial viability of the ECO process and equipment.

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9.0 List of Acronyms

ASN – Ammonium Sulfur Nitrates

CAVkit – Calibration Verification

CEMS – Continuous Emission Monitoring System

DBD – Dielectric Barrier Discharge

ECO – Electrolytic Catalytic Oxidation

ESP – Electrostatic Precipitator

QAQC – Quality Assurance Quality Control

QSIS – Quick Silver Inertial Separator

TVM – Total Vapor phase Mercury

WESP – Wet Electrostatic Precipitator

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